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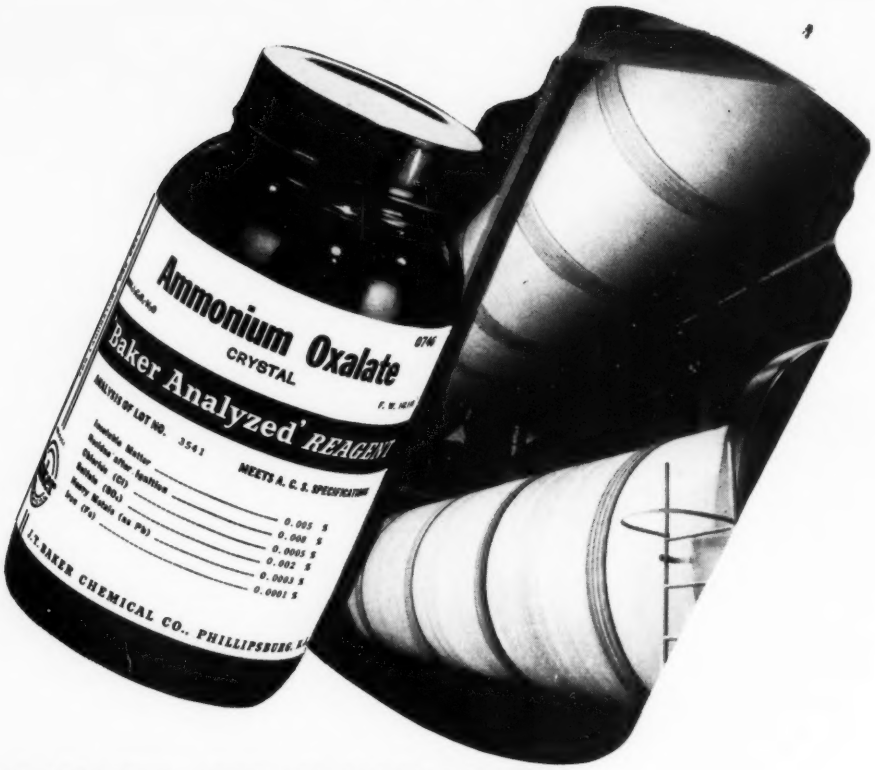

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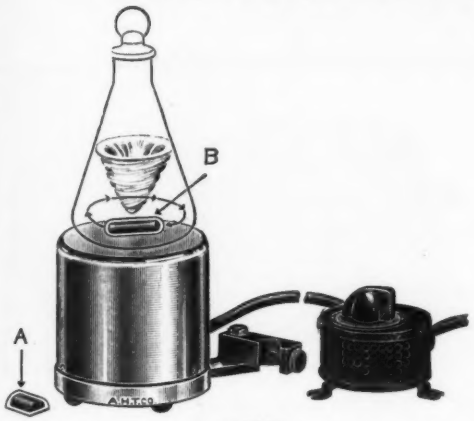

9235-R.

Fig. 1. Showing general stirring in a stoppered flask.

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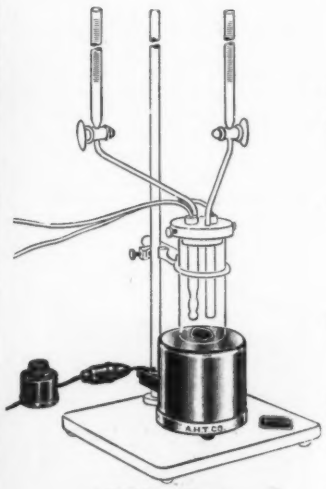

9235-R. Fig. 2

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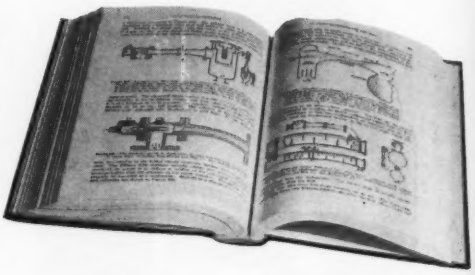

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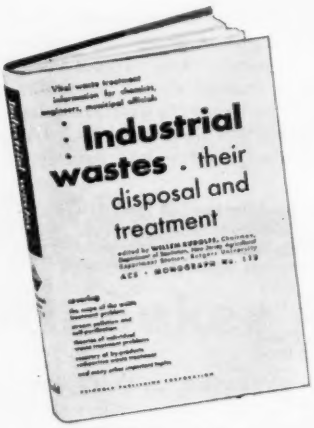

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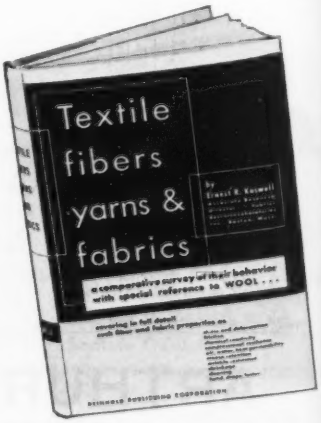

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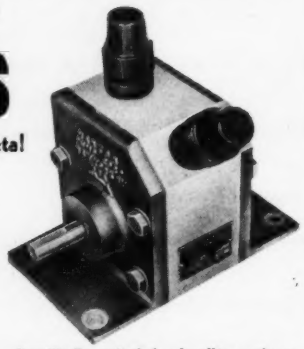

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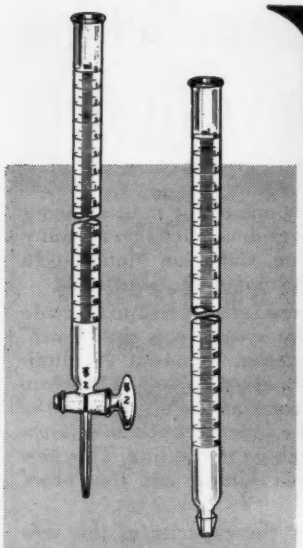

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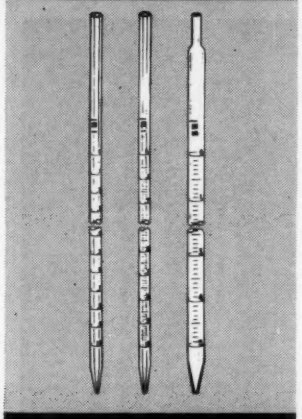

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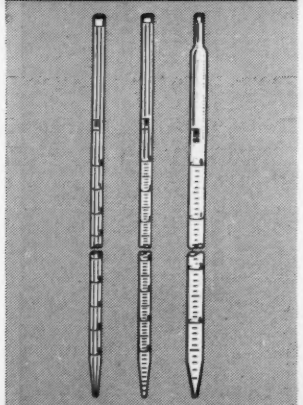

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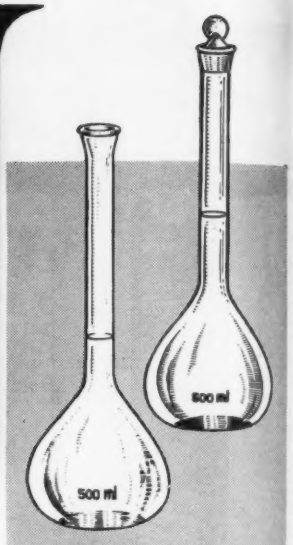

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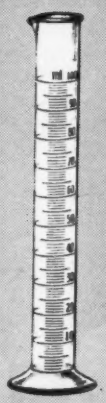

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		5	72	36.70	34.95	33.30	31.70
		10	72	41.70	39.70	37.80	36.00
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		2	72	38.35	36.50	34.75	33.10
		5	72	38.35	36.50	34.75	33.10
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		100	36	21.70	20.65	19.65	18.70
2050	Flask, Volumetric	10	36	30.45	29.00	27.60	26.30
		25	36	30.85	29.40	28.00	26.65
		50	36	32.10	30.55	29.10	27.70
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Newsweek, February 16, 1953

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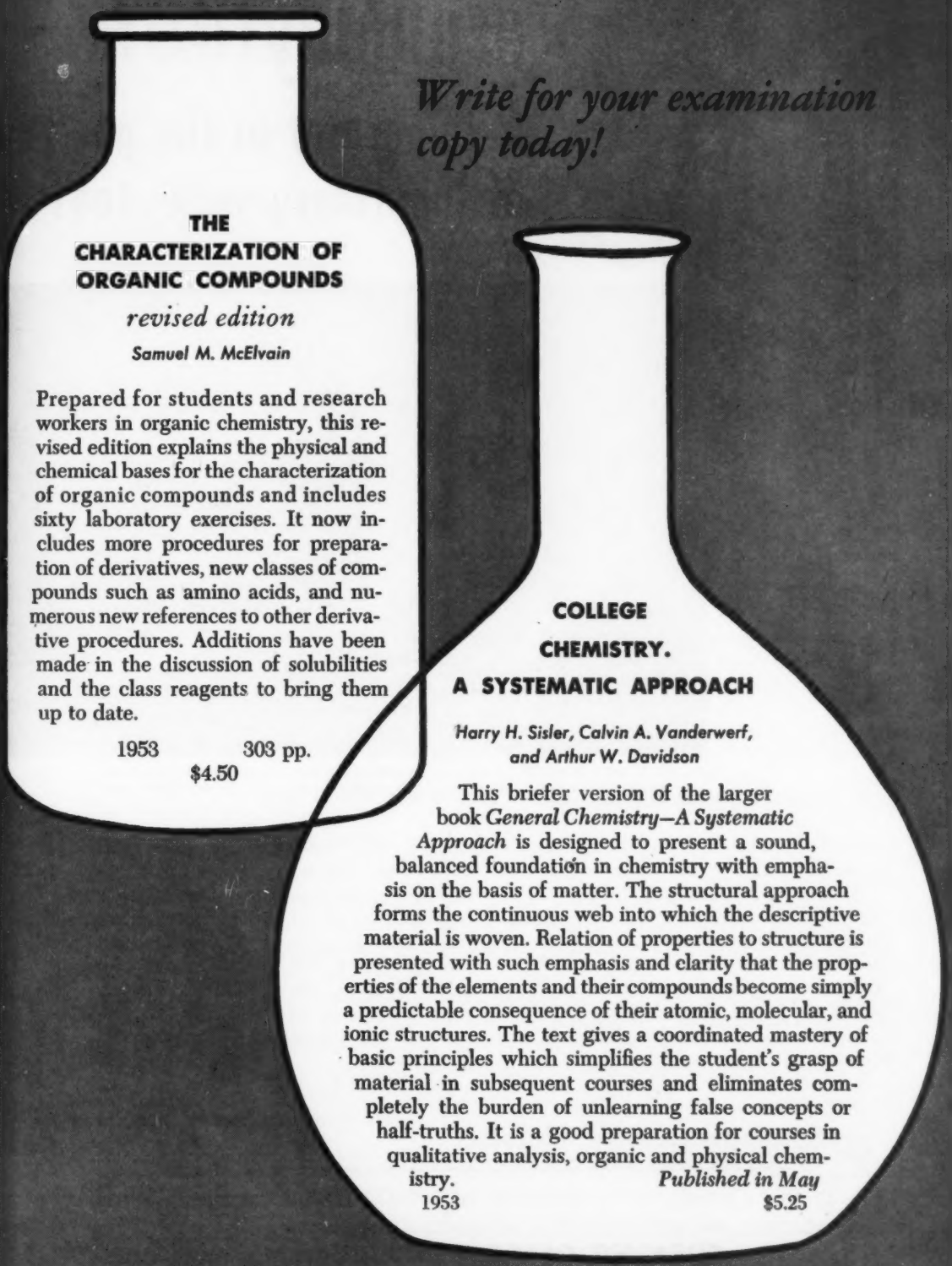

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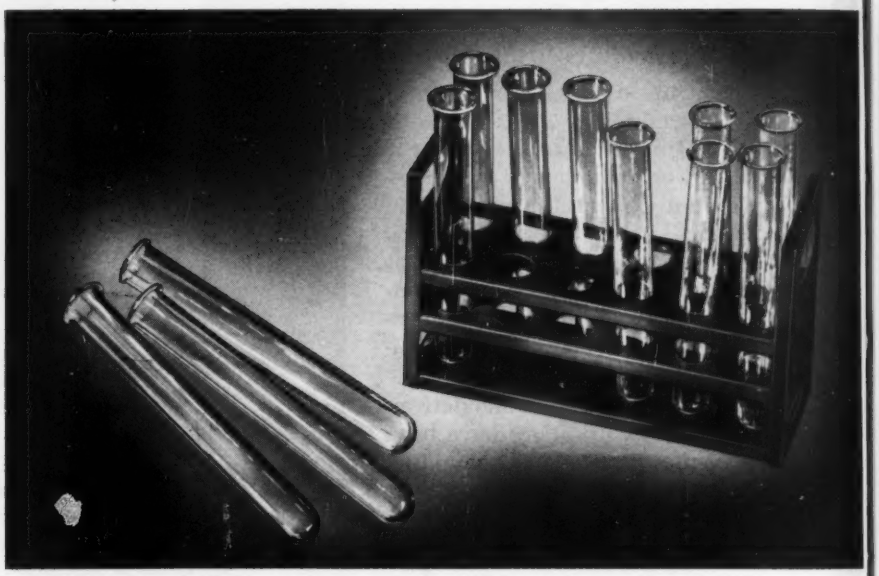

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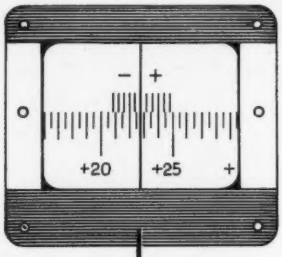

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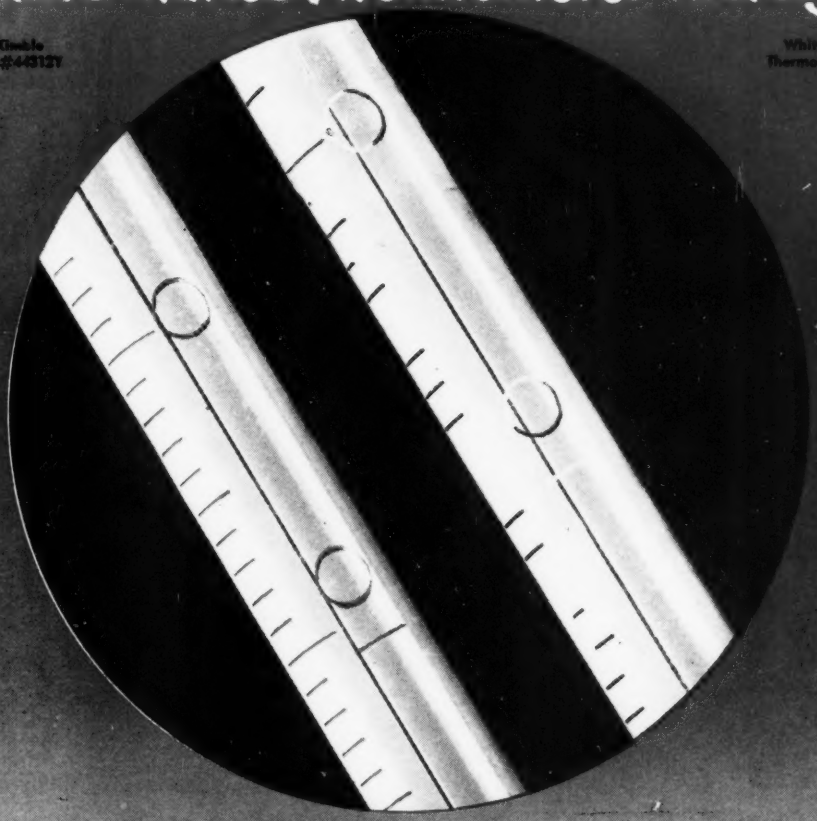

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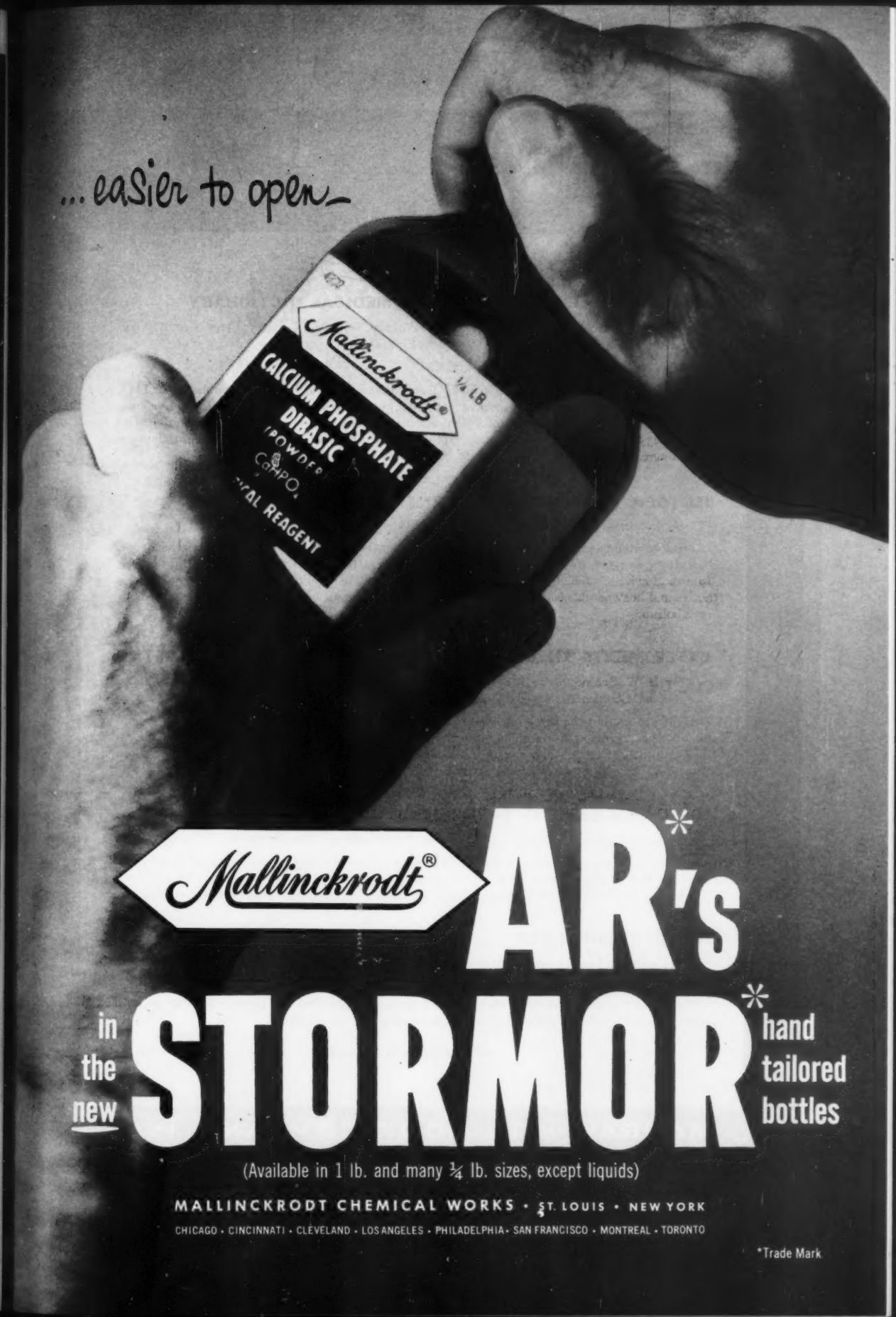
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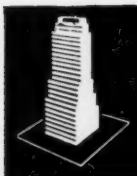

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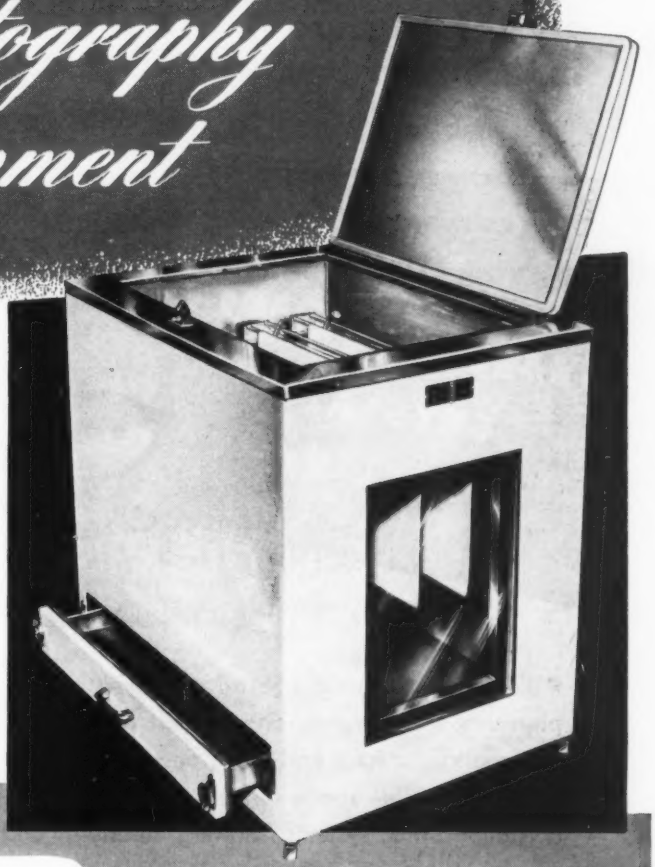

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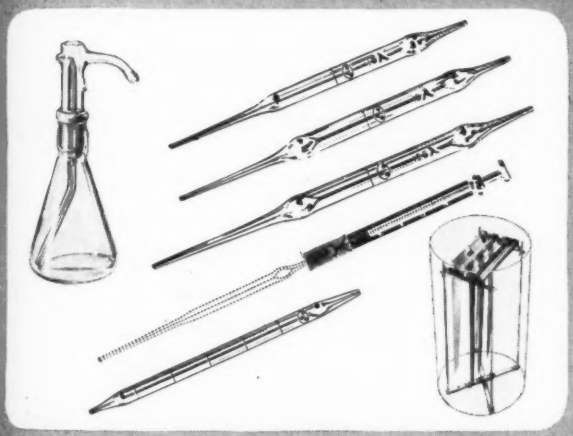

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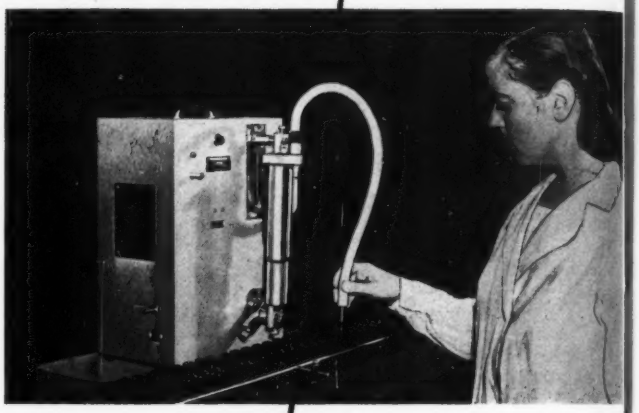

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(See page 313)

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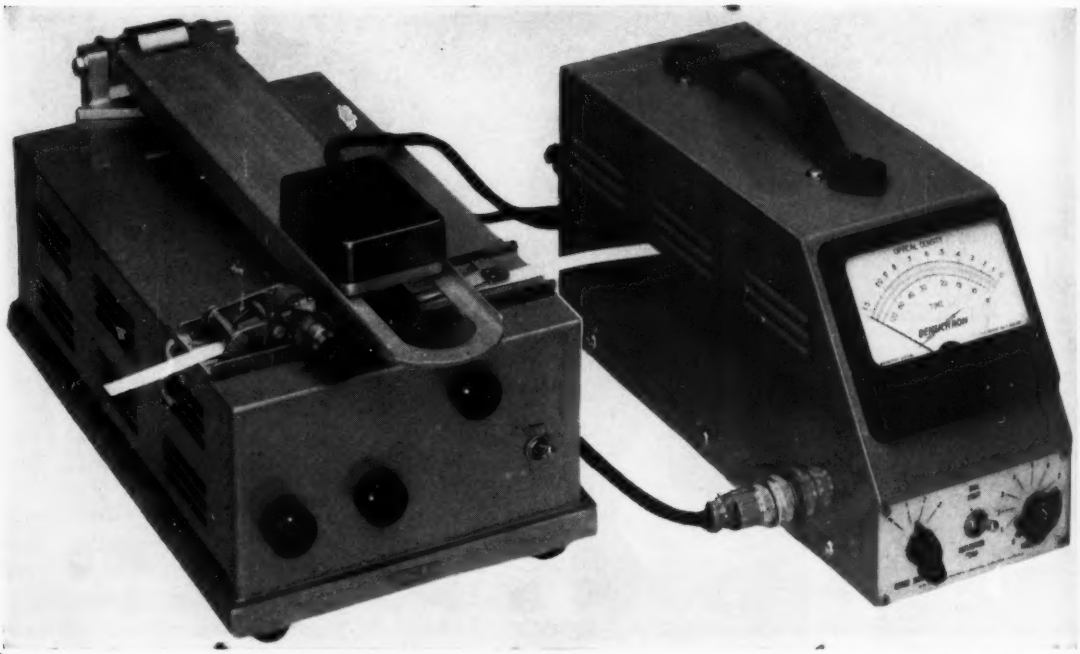

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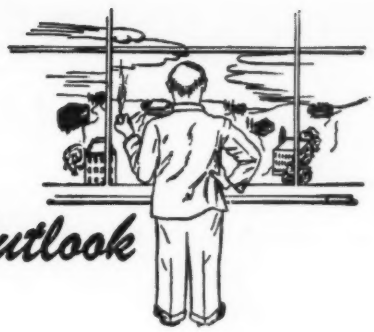

Editor's Outlook

It no longer surprises us to hear chemists and other technical folk extol the benefits of "general education." Perhaps it is the prick of conscience, perhaps it is a realization of the narrow restriction of their own early training. This may give some encouragement to those who believe that science is contributing more to the woe of the world than to its welfare. There are many who would welcome evidence to prove scientific training an opponent to liberal education.

It is therefore encouraging and refreshing to see a dyed-in-the-wool humanist state the case for the cultural value of science better than it has ever been stated by the scientists themselves.[1]

The author, Rhodes R. Stabley, is a professor of English at the State Teachers College at Indiana, Pennsylvania. The essence of his argument is that in the subject of literature, at least (which is certainly representative of the humanistic or liberal-arts tradition), it is difficult to avoid an authoritarian attitude, while true science, by its very nature, cannot possibly be authoritarian.

He quotes from a number of humanistic educators who maintain that a liberal arts education is especially necessary in these troublous times, and imply that science does nothing but increase our technological development and add to confusion. Then he comments:

> Not one indication that all subjects and areas of knowledge, from art to zoology, have moral and ethical, personality and citizenship contributions to make; only a clearly revealed conviction that science can have nothing to do with "ideals"; that indeed it is potentially dangerous to human values. The dangers have to be watched, the values have to be guarded by men nurtured in the liberal arts. Scientists—not to be trusted really; liberal arts men—the hope of the world.
>
> This attitude, as anyone knows who knows anything about the history of education, has long been standard stuff. It was standard stuff with the Greeks, who quite regularly put description ahead of observation; it was standard stuff with medieval thinkers, who loved to speculate on the number of angels able to stand on the head of a pin; it was standard stuff with the people who persecuted Galileo and reviled Newton; it has always been standard stuff with those who put words before facts, theories before operations, mind before senses. It is, alas, standard stuff in many quarters today. . . .
>
> "Great" literature can be especially dangerous because—backed up by the resounding and "immortal" names of Plato, Dante, Shakespeare, Milton, Blake, Shelley, Emerson, Poe—it carries impressive "authority." These men, it is so easy for us to say or imply (and for students to believe), spoke for all the ages; they were wise and profound and what they revealed was fitting not only for their own times and their own cultures, but for our times and our culture—and for all times and all cultures to come. Before we know it we are glibly talking about universal principles, eternal verities, and categorical imperatives, and being quite sure that we know exactly what we mean and that all men of good will and good sense must *agree* with what we mean. . . .
>
> Many institutions seek to control both students and teachers by a careful "selection" of authors and works. . . . In many institutions not all books studied are necessarily in line with the official attitude, but one must be extremely naïve to suppose that, except in the hands of a maverick teacher, these get anything like the sympathetic understanding and interpretation accorded to those safe within the main tendency. The special biases of many colleges—especially the smaller colleges, which, incidentally, are largely of the liberal arts type—are a fact well known to anyone who has made serious observations of higher education in this country. The fact should at least serve to challenge the notion that whatever bears the liberal arts label is therefore and thereby "liberalizing" in its effects on students. . . .
>
> All of which makes me considerably less sanguine than I used to be about the grand claims made automatically for the liberal arts-humanities tradition. All of which makes me feel slightly uncomfortable when the spokesmen for our tradition cavalierly dismiss the physical sciences as good only for physical ends. It would be good for them—and for all of us in education—at least to pause and consider these words of John Dewey: "It is impossible to say how much of the remedial suffering in the world is due to the fact that physical science is looked upon as being merely physical. It is impossible to say how much of the unnecessary slavery of the world is due to the conception that moral issues can be settled within conscience or human sentiment apart from consistent study of facts and application of specific knowledge in industry, law, and politics. . . . Each sign of disregard for the moral potentialities of physical science drafts the conscience of mankind away from concern with the interaction of man and nature which must be mastered if freedom is to become a reality. It diverts intelligence to anxious preoccupation with the unrealities of a purely inner life, or strengthens reliance upon sentimental outbursts of affection."[2]

Strong words—too strong, perhaps, for most of us, oriented as we are. But at least serving notice from one serious thinker that the sciences have more than purely physical contributions to make. Dewey is saying, indeed, that a workable morality can emerge *only* from a scientific attitude of mind. Now this attitude, as I understand it, eventuates in such habits as these:

(*Continued on page 283*)

[1] Stabley, R. R., "Character training: Liberal arts *vs.* science," *Bull. Am. Assoc. Univ. Profs.*, **38**, 625 (1952–53).

[2] Quoted from "Human Nature and Conduct" with permission of the publishers, Henry Holt and Co.

ISOTOPE STUDIES IN PHOTOSYNTHESIS[1]

J. A. BASSHAM,[2] A. A. BENSON and MELVIN CALVIN
University of California, Berkeley, California

THE net reaction of photosynthesis is usually written as

$$CO_2 + H_2O \xrightarrow{light} \{CH_2O\} + O_2$$

If we were to write under the arrow all the other elements essential to the various steps in the photosynthetic reaction, we would have to include phosphorus, nitrogen, sulfur, and a number of metal ions. Of these, phosphorus is thought to enter into photosynthesis as a reactant in a number of the steps involved in the reduction of carbon dioxide to organic compounds. The present discussion will be confined to the use of isotopic oxygen, hydrogen, carbon, and phosphorus.

OXYGEN

In any investigation of the mechanism of photosynthesis, one of the first questions that one would want to answer is whether the oxygen evolved in photosynthesis comes instantaneously from the carbon dioxide, the water, or both. These various possibilities are shown in the equations below.

$$CO_2 \rightarrow O_2 + [C] \qquad (a)$$

$$2H_2O \rightarrow O_2 + 2H_2$$
$$2CO_2 \rightarrow O_2 + 2CO \qquad (b)$$

$$2H_2O \rightarrow 2H_2 + O_2 \qquad (c)$$

$$H_2CO_3 \rightarrow O_2 + \{CH_2O\} \qquad (d)$$

$$2H_2O \rightarrow O_2 + 4[H]$$
$$4[H] + CO_2 \rightarrow \{CH_2O\} + H_2O \qquad (c')$$

The earliest proposals (*a*) suggested that the carbon dioxide is decomposed to oxygen and carbon, the carbon being later combined with water to give carbohydrates. In 1864 Berthelot proposed the reactions (*b*) in which water is decomposed to oxygen and hydrogen while carbon dioxide is decomposed to oxygen and carbon monoxide. These reactions are followed by a combination of carbon monoxide and hydrogen.

$$CO + H_2 \rightarrow \{CH_2O\}$$

In 1914 Bredig (*1*) suggested the decomposition of water only to oxygen and hydrogen (*c*), with subsequent reduction of the carbon dioxide by the hydrogen.

$$CO_2 + 2H_2 \rightarrow H_2O + \{CH_2O\}$$

In 1918 Willstätter and Stoll (*2*) proposed that carbonic acid is decomposed to give oxygen and an organic compound of the formaldehyde reduction level (*d*).

The proposal of a primary decomposition of water to hydrogen and oxygen (*c*) was modified by Thunberg (*3*) who suggested an intermolecular exchange between water and carbon dioxide (*c'*), and this theory was further formulated and supported by van Niel (*4*), who compared photosynthesis with the transfer of hydrogen atoms from other hydrogen "donors" to carbon dioxide in bacteria.

$$2H_2S \rightarrow 2S + 4[H]$$
$$4[H] + CO_2 \rightarrow \{CH_2O\} + H_2O$$

It can be seen that in (*a*) all of the oxygen comes from carbon dioxide, in (*b*) one half of the oxygen comes from carbon dioxide and one half from water, in (*c*) or (*c'*) all the oxygen comes from water, while in (*d*) all the oxygen comes from H_2CO_3, the hydration product of carbon dioxide. In the reaction

$$CO_2 + H_2O \rightleftarrows O{=}C(OH){-}OH$$

where the two hydroxyl groups are equivalent and in equilibrium with water, the contribution of the water to oxygen production will be $^1/_3$ if all the oxygen atoms of H_2CO_3 are equivalent, $^1/_2$ if the evolved oxygen comes from the hydroxyl groups only, and $^1/_4$ if one oxygen atom in the evolved oxygen originates in the C=O group.

By using a heavy isotope of oxygen, O^{14}, Ruben, *et al.* (*5*) tested these various possibilities in 1941. The unicellular algae, *Chlorella*, were allowed to photosynthesize in one experiment with O^{18}-labeled water and in another with O^{18}-labeled carbon dioxide, introduced in the form of dissolved $KHCO_3$ and K_2CO_3. In each case the evolved oxygen was collected and found to agree in isotopic content with the water and not with the carbon dioxide. Moreover, the isotopic oxygen content of the carbonate and bicarbonate was followed and it was verified that the time required for equilibration with the oxygen of the water was sufficiently slow to permit several collections of evolved oxygen before the difference between the O^{18} content of the bicarbonate-carbonate ions and that of the water became small. These results are summarized in Table 1. It can be seen that the O^{18} content of the evolved oxygen agrees in all cases with that of the water and not with that of the carbonate and bicarbonate. Therefore,

[1] Presented at the 122nd Meeting of the American Chemical Society, Atlantic City, September, 1952.

The work described in this paper was sponsored by the U. S. Atomic Energy Commission.

[2] Lt., USNR, Office of Naval Research Unit No. One, University of Calfornia, Berkeley. The opinions contained herein are the private ones of the writer and are not to be construed as official or reflecting the views of the Navy Department or the naval service at large.

only reaction (*c*) or (*c'*), in which the evolved oxygen originates in the water, can be correct.

There is another question regarding the photochemical evolution of oxygen during photosynthesis which can be answered by the use of isotopic oxygen. Warburg and co-workers, using the manometric method, followed the oxygen evolution by *Chlorella* while alternating short periods of illumination (down to one minute) with equally short periods of darkness (*6*). Under these conditions apparent quantum efficiencies approaching one molecule of oxygen evolved per quantum of light absorbed were observed. These workers proposed that in continuous photosynthesis one molecule of oxygen is actually being produced for each quantum of light absorbed but $^2/_3$ to $^3/_4$ of this oxygen is reabsorbed in a light-enhanced back reaction so that the net observed efficiency is one molecule of oxygen evolved per four quanta absorbed.

TABLE 1
Isotopic Ratio in Oxygen Evolved in Photosynthesis by Chlorella[a] (Ruben, Randall, Kamen, and Hyde(5))

Expt. No.	*Substrate*	*Time between dissolving $KHCO_3 + K_2CO_3$ and start of O_2 collection, min.*	*Time at end of O_2 collection, min.*	*Per cent O^{18} in $HCO_3^- + CO_3^{--}$* H_2O	CO_3^{--}	O_2
1	$KHCO_3$, 0.09 *M*	0	...	0.85	0.20	...
	K_2CO_3, 0.09 *M*	45	110	0.85	0.41[b]	0.84
		110	225	0.85	0.55[b]	0.85
		225	350	0.85	0.61	0.86
2	$KHCO_3$, 0.14 *M*	0	...	0.20	...	...
	K_2CO_3, 0.06 *M*	40	110	0.20	0.50	0.20
		110	185	0.20	0.40	0.20
3	$KHCO_3$, 0.06 *M*	0	...	0.20	0.68	...
	K_2CO_3, 0.14 *M*	10	50	0.20	...	0.21
		50	165	0.20	0.57	0.20

[a] The volume of evolved oxygen was large compared with the amount of atmospheric oxygen present at the beginning of the experiment.
[b] Calculated values.

This proposal presumes that there is a rapid equilibrium between the measured oxygen gas and the oxygen within the cell wall; otherwise, the slopes of the curves of oxygen evolved versus time during the one-minute intervals, upon which the conclusions are based, are meaningless.

If these proposals and assumptions are correct and if the gaseous oxygen in contact with the cell suspension is labeled with O^{18}, then the rate of entry of O^{18} into the water and into the organic compounds within the cell should be greatly enhanced in the light as compared with this rate in the dark (*7*). Brown, Nier, and Van Norman (*8*) have performed just such an experiment and found no light-enhanced increase in the rate of disappearance of O^{18} from the gas phase. These workers applied a new technique in measurement of isotopic oxygen in that they measured the isotopes continuously using a recording mass spectrometer. Gas from a small Warburg vessel containing algae and labeled oxygen was continuously allowed to leak into the spectrometer. A typical record of O_2 of mass 32 and of mass 34 ($O^{18}O^{16}$) is reproduced in Figure 1. The O^{18} (initially only in the gas phase) is absorbed at a constant rate, while O^{16}, produced by photosynthesis, increases in the light. O^{18} uptake is unaffected by light and dark periods and would suggest that respiration is independent of photosynthesis.

The above experiments provide information regarding the origin of the evolved oxygen but tell us little of the intermediates involved in the transformation of water to oxygen and hydrogen. Dorough and Calvin (*9*) have investigated the possible increase in O^{18} (in the form of epoxides) in xanthophyll pigments during photosynthesis. *Chlorella* were suspended in water containing 4 per cent O^{18} enriched oxygen. Bicarbonate was added to the suspension which was divided into two equal portions. One portion was allowed to photosynthesize in the light while the other portion was kept in the dark. Gas exchange was followed with a Warburg apparatus. After five hours the algae were centrifuged and killed. The xanthophylls were partially purified by extraction and chromatography, their oxygen was converted to CO, and the latter was analyzed with a mass spectrograph. The results are shown in Table 2.

The authors were limited by the fact that they had only 50 ml. of labeled water and the O^{18} concentration was only 4 per cent. Isolation of the furan and epoxide carotenoids gave about 5 mg. of these compounds together with an equal amount of steroids. Further purification would have reduced the amount of material

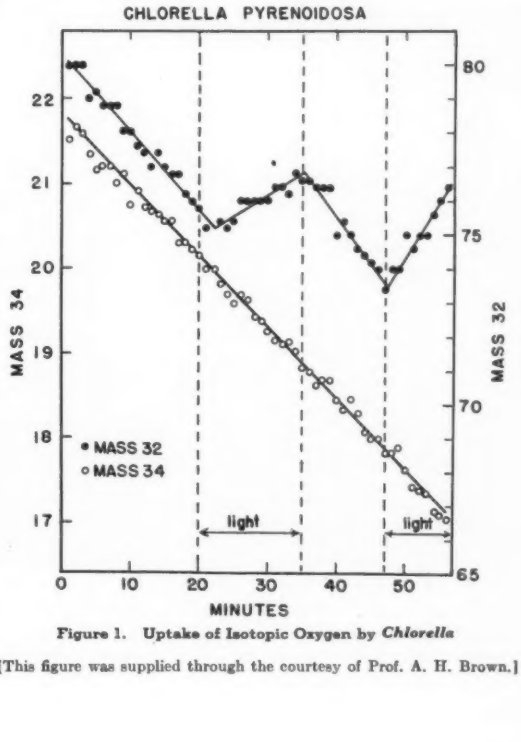

Figure 1. Uptake of Isotopic Oxygen by *Chlorella*

[This figure was supplied through the courtesy of Prof. A. H. Brown.]

TABLE 2 (9)
Isotopic Ratio in Oxygen Found in Xanthophyll from Chlorella Exposed to H_2O^{18} (Dorough and Calvin)

	Sample	O^{18} %
1.	Original H_2O	4.0
2.	Water in equilibrium with algae: light run	3.01
3.	Water in equilibrium with algae: dark run	3.06
4.	CO sample from xanthophyll fraction of photosynthesized (5 hr.) algae (50% carotenoid)	0.245
5.	CO sample from xanthophyll fraction of algae kept in the dark (40% carotenoid)	0.233
6.	Normal H_2O	0.204

to a point where enough would not be left for mass-spectrographic analysis. Therefore, the impure sample had to be used, which resulted in an estimated tenfold dilution of the O^{18}, so that the expected increase of O^{18} content of the carotenoids in the light would be at most 0.4 per cent as compared with a normal content of 0.2 per cent. The accuracy of the mass-spectrographic method is sufficient to allow one to say that the analyzed sample from the light-exposed algae actually contained more O^{18} than that from the "dark" algae in this one experiment but no further conclusions are justified at present. The results do suggest that the experiment would be worth repeating with higher O^{18} enrichment of the water and a larger quantity of algae.

HYDROGEN

Another way in which one might study the decomposition of water during photosynthesis and perhaps the transfer of hydrogen to other molecules is found in the use of isotopic hydrogen. Two such isotopes are available: deuterium, H^2, a stable isotope; and tritium, H^3, an emitter of low energy β particles. Since these isotopes differ in mass from H^1 by a factor of 2 and 3, respectively, there is a possibility for a much greater difference in the reaction and diffusion rates among these isotopes than would be expected among isotopes of higher atomic weight (such as O^{16} and O^{18} or P^{31} and P^{32}).

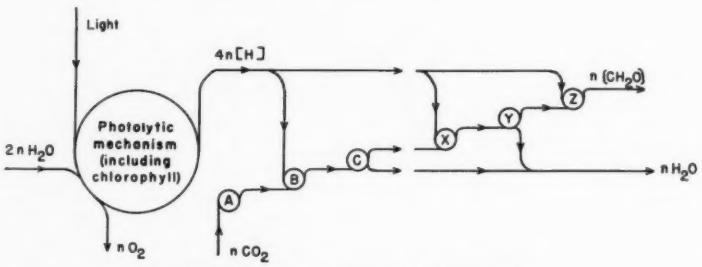

Figure 2. Schematic Diagram of Photosynthesis

In 1942 Norris, Ruben, and Allen (*10*) studied the photosynthesis of *Chlorella* in water containing tritium. These workers found no incorporation of tritium into chlorophyll during photosynthesis. In 1948, Calvin and Aronoff (*11*) performed similar experiments with deuterium but again the results were negative. Although these results may mean that chlorophyll does not serve as a reversible hydrogen donor, they could be explained in a number of other ways. For example, the chlorophyll hydrogen involved in photosynthesis might be an enolizable hydrogen which would exchange with water hydrogen, thus giving the same result in the dark as in the light.

Another possibility is that the chlorophyll extracted from plants is the oxidized form of the redox couple involved in the photochemical hydrogen transfer, so that no labeling would be observed even if the reduced form in the plant does become labeled. Since one reduced form of chlorophyll, 3,4-dihydrochlorophyll *a*, or bacteriochlorophyll (*12*) can be isolated from photosynthetic bacteria, it might be profitable to repeat the labeled-hydrogen experiments with these organisms.

CARBON

By far the greater part of the tracer studies of photosynthesis have been carried out with isotopic carbon. In 1939 Ruben and co-workers (*13*) reported the results of dark fixation of carbon dioxide labeled with C^{11}. Evidence was found for the incorporation of carbon dioxide via carboxylation reactions. This type of carbon fixation is now known to be characteristic of the incorporation of carbon dioxide by certain reversible reactions of respiration which are quite similar for both photosynthetic and non-photosynthetic tissue. The short half life of C^{11} placed a severe limitation on the type of experiments that could be carried out with this isotope. The increased availability of C^{14} after 1945 made possible more elaborate experiments.

In the early experiments with C^{14} the dark fixation of radioactive carbon dioxide by algae was studied. It soon became apparent that the incorporation of carbon dioxide in the dark by algae was greatly dependent on the previous condition of light or dark, the uptake of carbon dioxide being 20 times as much in the dark immediately after illumination as in the dark without preillumination (*14*).

The processes of photosynthesis are indicated schematically in Figure 2. The left half of this diagram represents the absorption of light by chlorophyll and the transfer of this energy, resulting in the photolysis of water and the formation of oxygen and hydrogen, the latter in the form of some unknown reducing agent which acts as either a hydrogen carrier or an electron donor. The overall reaction for this process may be written

$$2H_2O + \text{light} \xrightarrow[\text{chlorophyll}]{} 4[H] + O_2$$

The right portion of the diagram represents the reduction of carbon dioxide to carbohydrate or other organic end products of photosynthesis through a number of unspecified intermediates. The net reaction for this process can be written

$$CO_2 + 4[H] \rightarrow \{CH_2O\} + H_2O$$

The separation of the process of photolysis of water

from the reduction of carbon dioxide is based on several kinds of evidence, including the result of studies of the origin of the evolved oxygen already discussed. Further evidence for this separation was found in the enhanced carbon dioxide dark fixation following preillumination. In order to show that the latter effect was similar to the reduction of carbon during photosynthesis, it was necessary to compare the intermediate products formed in the two cases and this in turn involves labeling, analysis, and identification of compounds A, B, C, etc., in Figure 2 (*15*). When this comparison was made, using the techniques that will be described later, the products formed were found to be quite similar for photosynthesis and pre-illuminated dark fixation and very different for dark fixation not pre-illuminated.

It soon became clear that for the many experiments which would be required, it would be necessary to have available in the laboratory a constant source of plant material with properties which would vary as little as possible from time to time. For this purpose the conditions of growth would have to be easily reproducible. These requirements are best satisfied by the unicellular algae, such as *Chlorella* and *Scenedesmus*. In our laboratory these algae are grown in flasks mounted on a shaker and immersed in a thermostated bath and illuminated from below. The suspension of algae is withdrawn every other day, 10 per cent of the volume being left in the flask as an inoculum to which fresh inorganic nutrient solution is added. The growing algae are aerated with carbon dioxide-enriched air. We have, in effect, continuous cultures which have been maintained for four months or more. The harvested algae are centrifuged, washed, and resuspended in a flat illumination vessel, shown in Figure 3. A stream of carbon dioxide-enriched air is bubbled through the suspension during illumination until the algae reach a steady state of photosynthesis. At the start of a normal photosynthesis experiment, a suitable amount of C^{14}-labeled sodium bicarbonate is injected into the suspension and photosynthesis is allowed to proceed for a predetermined number of seconds, after which the large stopcock at the bottom of the flask is opened and the algae run rapidly into boiling ethanol to stop enzymatic reactions.

In the case of pre-illumination experiments, the procedure is the same except that at the moment of injecting the radiocarbon, the suspension is placed in the dark.

If a study of carbon reduction as a function of time is being made (in either light or dark) aliquots of the suspension are allowed to run from the flask into the alcohol at measured time intervals. In some experiments, leaves of higher plants, such as soybean or barley, are used, in which case a flat illumination vessel with a detachable face is used. At the start of the experiment labeled carbon dioxide is admitted to the chamber and after the exposure the reaction is stopped by removing the face of the vessel and plunging the leaf into alcohol.

After the plants have been killed and extracted by 80 per cent alcohol in water and then by 20 per cent alcohol in water, the radioactivity incorporated into non-volatile compounds is determined by removing a small aliquot of the extracted material and mounting on an aluminum plate for counting with a Geiger-Müller counter. The insoluble material is resuspended and similarly counted. It was found that in short periods of photosynthesis nearly all the fixed radiocarbon is in the soluble extract, as is seen in Figure 4.

The analysis of the radioactive compounds can then be carried out. Preliminary fractionation of the products of carbon dioxide fixation indicated a very complex pattern even in rather short periods of photosynthesis. Fortunately, a method which had been developed by Consden, Gordon, and Martin (*16*) for the analysis of amino acids proved applicable to the analysis of the products of carbon dioxide fixation. This method, two-dimensional paper chromatography, was combined with radioautography to give a relatively simple and complete method of separating and detecting the C^{14}-labeled products. The extracts are combined and concentrated to a small volume. An aliquot of this material is then run onto a small area in the corner of a large square sheet of filter paper and dried with an air stream. The chromatogram is then developed in the usual way (*17*) giving a two-dimensional distribution of all the extractable, soluble compounds from

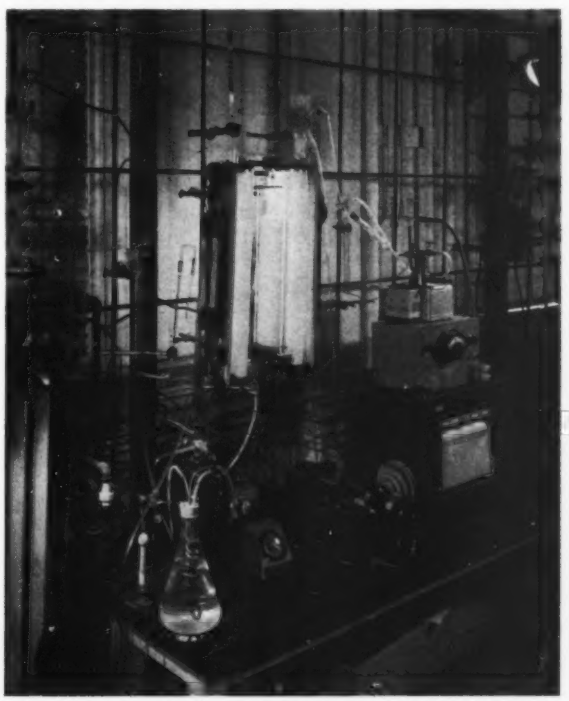

Figure 3. Apparatus for Exposing a Suspension of Algae to $C^{14}O_2$ During Photosynthesis

This experimental arrangement includes flat illumination vessel, fluorescent lights, gas circulating system, and continuous recording system for measuring radioactivity of $C^{14}O_2$.

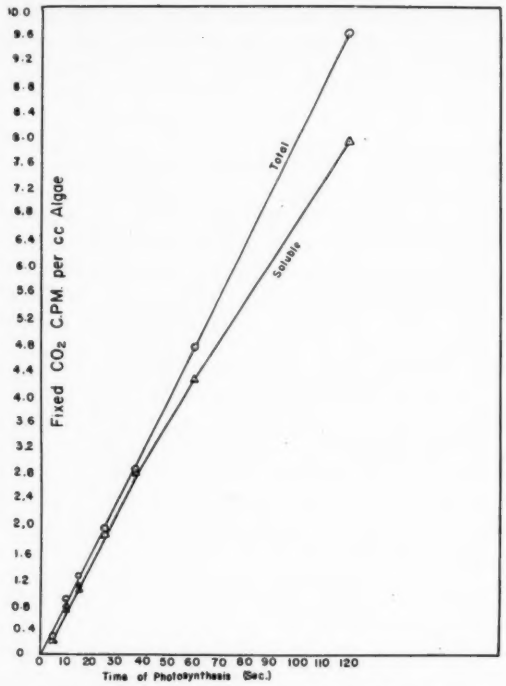

Figure 4. Comparison of C^{14} Incorporated into All Plant Constituents during Photosynthesis with that Incorporated into Soluble Compounds

The soluble compounds are those extracted by 80 per cent ethanol in water and 20 per cent ethanol in water.

the plant except for those which were volatile or unstable under the conditions of analysis.

A sheet of X-ray film is then placed in contact with the chromatogram. The C^{14} in the labeled compounds emits beta particles which expose the film at the positions of the compounds. In this way we are able to locate precisely those compounds in which we are interested. A radioautograph of the carbon-fixation products of 60 seconds photosynthesis by *Chlorella* is shown in Figure 5. This photographic film corresponds to a chromatogram on which the origin is near the lower right corner. The first solvent used to develop the chromatogram was phenol-water, running from right to left, and the second solvent was butanol-propionic acid-water, running from bottom to top.

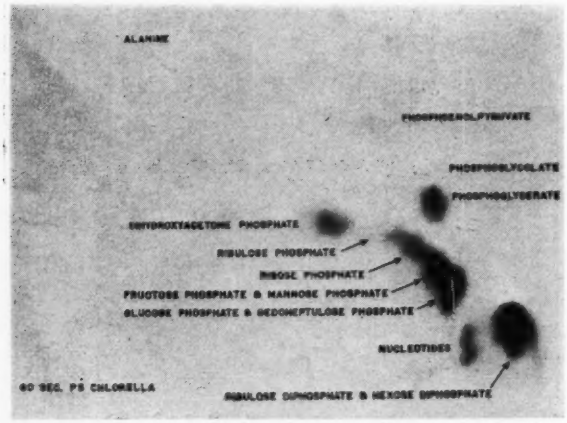

Figure 5. Radiogram of Soluble Compounds Formed during 60 Seconds Photosynthesis with *Chlorella*

Cells killed at end of experiment by 80 per cent ethanol in water at room temperature. Light intensity was 5000 foot candles from each side of the illumination vessel.

After the analysis of the extract by paper chromatography and detection of the radioactive compounds by the radioautograph, the next major problem is the identification of these compounds.

The first step in identifying the compounds corresponding to darkened areas of the X-ray film ("spots") is to establish the position of a number of known compounds in the particular chromatographic system used. This is usually accomplished by chromatographing known unlabeled compounds on the paper chromatogram by means of suitable chemical spray tests. These chemical sprays (which are sometimes followed by heating of the paper or other development methods) cause a color to develop with the compound, thus allowing it to be seen. Usually, different classes of compounds require different spray tests. For example, amino acids are detected with ninhydrin spray (*16*), phosphates with ammonium molybdate (*18*, *19*), ketoses with naphthoresorcinol (*20*), and aldoses with aniline hydrogen phthalate (*21*). Carboxylic acids often can be located by means of a pH indicator spray (*22*). Occasionally, when synthetically labeled radioactive compounds are available, they are chromatographed and detected by means of their radioactivity.

After the positions of a number of compounds have been determined in a given solvent system, a map is prepared of these positions.

The second step in identifying a new compound is to compare its chromatographic position with that of the compounds on the map. Such a comparison often provides an excellent clue to the nature of the compound. Further identification depends on a number of observations in which the compound is eluted from the paper, treated chemically, and the resulting material chromatographed a second time to determine whether or not the supposed chemical transformation has taken place in the radioactive compound.

For final identification, the unknown radioactive material is chromatographed together with an authentic sample of the suspected substance and complete coincidence of the radioactivity, as defined by the spot on the X-ray film, with the known material, as defined by some colored or otherwise visible product, is observed. This type of co-chromatogram, when carefully interpreted, provides an unequivocal identification since not only the positions of the radioactive and colored spots must coincide but the fine irregularities around the edge of the spot caused by the structure of the paper must exactly coincide also. The latter provide almost as unique an identification as a fingerprint.

Through the use of these methods of identification a

number of the early compounds formed during photosynthesis have been identified, as shown in Figure 5. The radioactivity incorporated in a given compound can then be measured by cutting out the area of paper containing that particular compound, as indicated by the spot on the X-ray film, eluting the compound from the paper, and mounting on a plate for counting.

A faster but slightly less accurate method of determining radioactivity consists of counting the spot directly on the paper by placing a thin-window Geiger tube directly on the spot. The fraction of the total radioactivity of the spot registered by the counter in this case is about one-third that obtained from a plate of the same material and is fairly constant for all C^{14}-labeled organic compounds.

When this was done, it was found that in very short times the radioactivity is incorporated in only a few compounds. The result of 10 seconds incorporation of C^{14}-labeled bicarbonate by *Scenedesmus* is shown in Figure 6. A considerable part of what is known about the path of carbon reduction during photosynthesis is indicated by this one chromatogram. First of all, it is seen that nearly all the radiocarbon is found in phosphorylated compounds. This is indicative of the important role phosphorus plays in carbon reduction in photosynthesis.

Second, over half the radioactivity is found in one compound—phosphoglyceric acid. At 5 seconds exposure more than 80 per cent of the radioactivity is found in PGA. If the percentage of total radioactivity found in PGA is given as a function of time of exposure, we find that in a series of experiments with different exposure times, this function approaches 100 per cent at zero time. Thus, PGA appears to be the first product of carbon dioxide reduction that we can see by this method of analysis, under these conditions of photosynthesis.

If we look at the other compounds that are labeled in this short time, we find among them a triose phosphate, hexose monophosphates, and hexose diphosphates. These compounds suggest the formation of hexoses from phosphoglyceric acid via the well-known reversible reactions of glycolysis.

Finally, we should note for later reference the early appearance of pentose and heptose phosphates.

Having devised a means for following the incorporation of carbon dioxide into different compounds, it becomes equally important to find a way of observing the rate of labeling of individual carbon atoms of each compound. Thus, if phosphoglyceric acid is formed by a carboxylation reaction, we would hardly expect that the radiocarbon would be uniformly distributed among the three carbon atoms of this molecule after short exposures to labeled carbon dioxide. When a chemical degradation of glyceric acid, obtained from hydrolysis of phosphoglyceric acid, is carried out, nearly all the radiocarbon is found in the carboxyl position when the period of photosynthesis with radiocarbon has been only 5 seconds. After longer periods, more of the total radioactivity is found in the other two positions. In Table 3 several examples of these degradations are shown. From these results we find support for several proposals regarding the specific steps in carbon dioxide reduction.

It appears that PGA is formed by a carboxylation of a two-carbon compound and that this carbon dioxide acceptor is labeled at an equal rate between the two positions. Furthermore, this rate is quite slow compared with the rate of the primary carboxylation, which indicates that the two-carbon compound is not formed

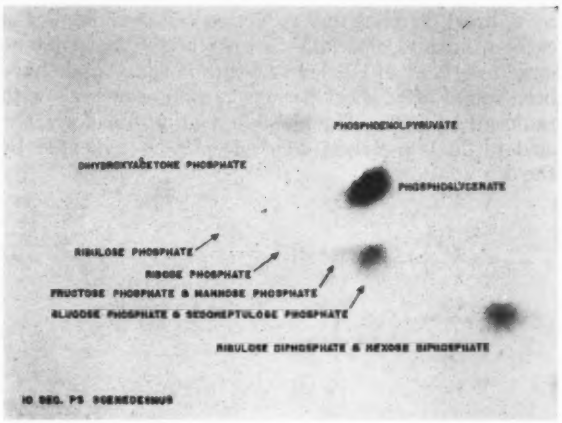

Figure 6. Radiogram of Soluble Compounds Formed during 10 Seconds Photosynthesis with *Scenedesmus*

Other experimental conditions are the same as for Figure 5.

TABLE 3
Distribution of Radiocarbon in Compounds Labeled during Photosynthesis

Compound		Conditions: Preillum 2 min. dark	4 sec. PS (Photosynthesis)	15 sec. PS	15 sec. PS	40 sec. PS	60 sec. PS
Glyceric acid	COOH	96.0	87.0	56	49	...	44
	CHOH	2.6	6.5	21	25	...	30
	CH_2OH	1.7	6.8	23	26	...	25
Glycolic acid	COOH	...	49.0	50 ± 5	...	47	
	CH_2OH	...	52.0	50 ± 5	...	53	
Hexose (from sucrose)	1 C				24		
	2 C				25		
	3 C				52		
	4 C						
	5 C						
	6 C						

by a direct coupling and reduction of two molecules of carbon dioxide, especially in view of the fact that no large reservoirs of labeled two-carbon compounds have been found after short periods of photosynthesis with radiocarbon (*23*). The possible relationship of glycolic acid to the two-carbon CO_2 acceptor is indicated by the degradation data.

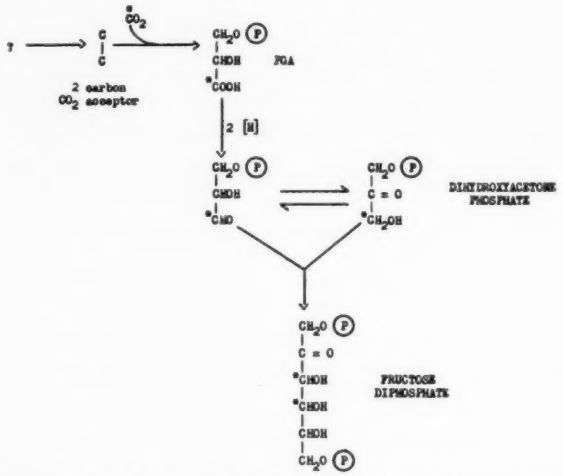

Figure 7. Path of Carbon from CO_2 to Hexose during Photosynthesis

The agreement between the degradation for sucrose and that for phosphoglyceric acid provides further evidence for the formation of hexoses by the reversible reactions of glycolysis. If two molecules of PGA were reduced and condensed to hexose, we would expect carbons 3 and 4 of the hexose to correspond in labeling to the carboxyl of the PGA, and carbons 1, 2, 5, and 6 of the hexose to correspond to the α and β carbon atoms of PGA, as indeed they do.

These conclusions are summarized in Figure 7, in which the path of carbon from free carbon dioxide to hexose is indicated.

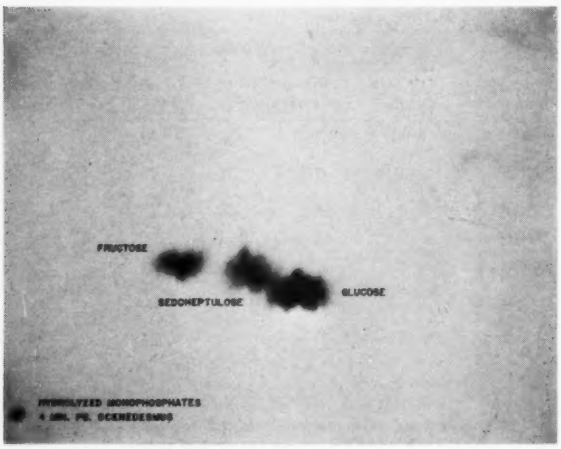

Figure 8. Radiogram of Sugars Obtained by Phosphatase Hydrolysis of the "Hexose Monophosphate" Area from a Chromatogram of Soluble Compounds Formed in 4 Minutes Photosynthesis with *Scenedesmus*

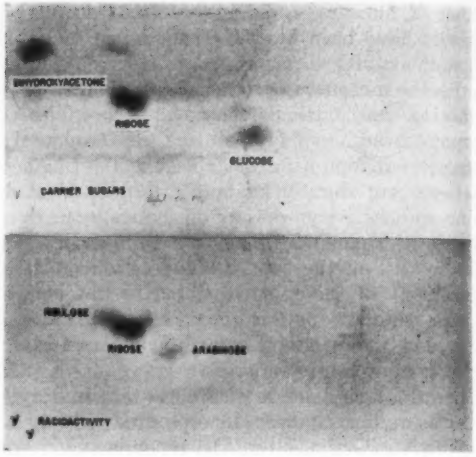

Figure 9. Co-chromatography of Unlabeled Authentic Sugars with Labeled Sugars Obtained by Phosphatase Hydrolysis of the "Ribose Phosphate" Shown in Figure 5

There remains the problem of the regeneration of the two-carbon CO_2 acceptor. The reasons for not believing it to be formed by direct coupling and reduction of carbon dioxide have been indicated. Moreover, in short exposures of the plant to radioactivity (30 sec. or less), it is possible to account for virtually all the carbon fixed (95 per cent or more) in the form of soluble compounds which can be chromatographed and detected by radioautography. Since the α and β carbon atoms of PGA (and hence of the 2-carbon CO_2 acceptor) are appreciably labeled in these short times, this means that the CO_2 acceptor is probably derived from some of the labeled compounds that are seen in these experiments.

In order to appraise this possibility, a more detailed study of the phosphates seen in Figure 5 is necessary. If one takes each of these compounds or groups of compounds and subjects it to a phosphatase enzyme and then chromatographs the resulting compounds, one obtains chromatograms of the phosphate-free compounds which often are more easily separated and identified than the original phosphates.

Figures 8, 9, 10, and 11 show such chromatograms. The hexose and heptose monophosphate areas from a chromatogram similar to that seen in Figure 5 have been treated with phosphatase and the resulting free sugars chromatographed. When this is done for all the phosphate esters on the chromatogram, there are found, in addition to those compounds already mentioned, two sugars which do not have any known connection with the glycolysis scheme. These are sedoheptulose and ribulose, seven- and five-carbon ketoses, respectively. Since each of these sugars, in the form of phosphates, becomes labeled in very short times, one is tempted to assign to them a role in the regeneration of 2-carbon compounds. One such scheme which has been proposed (*24*) is seen in Figure 12.

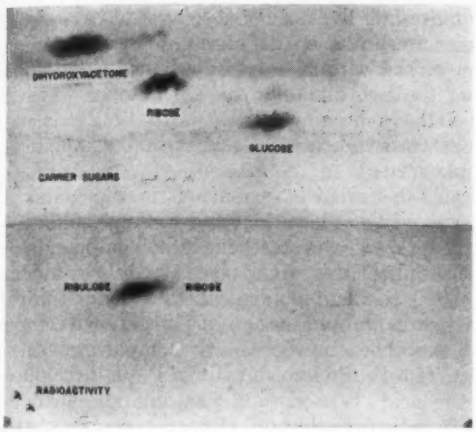

Figure 10. Co-chromatography of Unlabeled Authentic Sugars with Labeled Sugars Obtained by Phosphatase Hydrolysis of the "Ribulose Phosphate" Shown in Figure 5

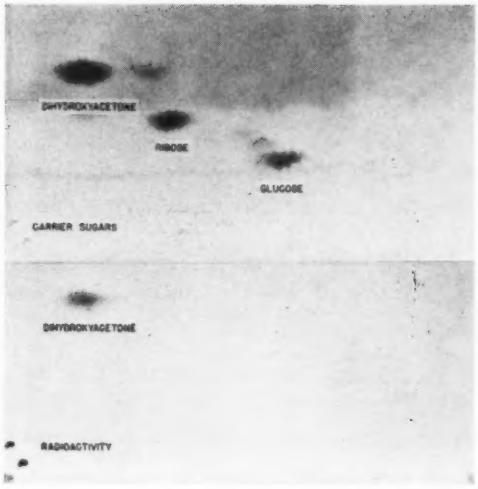

Figure 11. Co-chromatography of Unlabeled Authentic Sugars with Labeled Sugars Obtained by Phosphatase Hydrolysis of the "Dihydroxyacetone Phosphate" Shown in Figure 5

In this scheme, for each two molecules of the C_3 compound (PGA) formed by carboxylation of two C_2 compounds, one is used in hexose synthesis and one is further carboxylated to give a four-carbon compound. This C_4 compound is reduced and condensed with a C_3 to give a C_7 sugar which then splits to C_2 and a C_5 sugar. The latter splits to C_3 and C_2, completing the cycle. The net reaction is the reduction of three carbon dioxide molecules to one-half a hexose molecule for each turn of the cycle. The driving force which turns this cycle would be, of course, the reducing agent, formed by the photolysis of water, which reduces carboxylic acids to aldehydes and alcohols at appropriate points.

The missing link in this scheme is the lack of appearance of four-carbon acids and sugars in experiments thus far. However, some malic and aspartic acids have been found labeled in fairly short times and, while these acids are not believed to be intermediates (*23*), they may well be indicators of actual intermediates. Perhaps the actual four-carbon intermediates, if there are any, are present in such small concentrations that they are not detected even by the sensitive tracer method.

One experiment which indicates that this may be the case was a ten-second exposure of soybean leaves to $C^{14}O_2$. The distribution of radiocarbon in the soluble compounds was as follows: PGA, 32 per cent; sedoheptulose monophosphate, 24 per cent; fructose monophosphate, 18 per cent; glucose monophosphate, 6 per cent; triose phosphate 8 per cent; pentose diphosphate and monophosphate, 9 per cent; phosphoenol pyruvic acid, 3 per cent. This result may indicate that the four-carbon carboxylation product is so rapidly reduced and condensed with a three-carbon compound that the compound which actually indicates this carboxylation is sedoheptulose monophosphate.

Other explanations of the early appearance of sedoheptulose are quite conceivable. One such possibility is that the four-carbon fragment arises from the splitting of hexose to a C_2 fragment and a C_4 fragment. In any event, further experiments, in which constantly improving chromatographic methods are combined with more degradations and more accurate kinetic experiments, should help answer the question of the actual mechanism of carbon reduction in plants.

PHOSPHORUS

The importance of phosphorus in the processes of carbon reduction during photosynthesis is apparent from the foregoing discussion. While most of the tracer investigations of phosphorylated intermediates in carbon reduction employed C^{14}, nevertheless P^{32} has served as

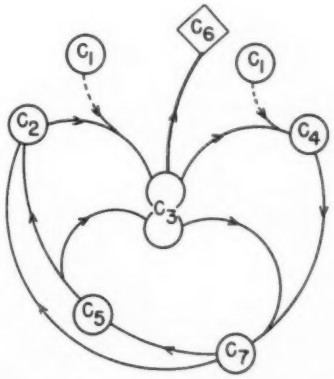

Figure 12. Proposed Carbon Cycle for Regeneration of Two-Carbon CO_2 Acceptor

a valuable auxiliary tool for this purpose. Sometimes both C^{14} and P^{32} were administered in the same experiment. The radioautographs of the chromatographed products of such an experiment are shown in Figures 13 and 14. In this case *Scenedesmus* were sus-

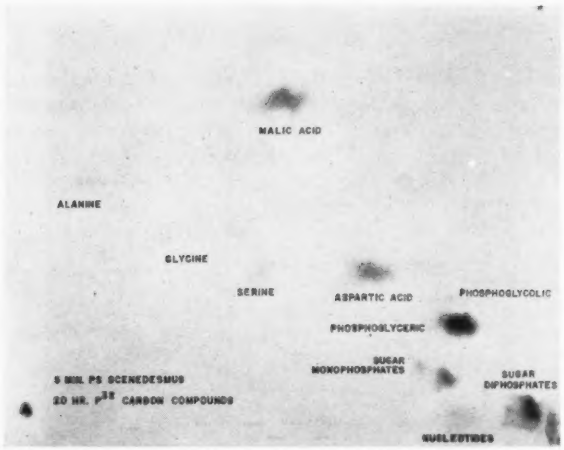

Figure 13. C^{14}-Labeled Compounds Formed During 5 Minutes Photosynthesis with C^{14}-Labeled Bicarbonate with *Scenedesmus* Which Had Been Exposed to P^{32} for 20 Hours

This radiogram was obtained by exposing X-ray film to the chromatogram after essentially all the P^{32} had decayed.

pended in a solution containing radioactive phosphate for 20 hours and then were allowed to photosynthesize for five minutes with C^{14}-labeled bicarbonate before killing. Since the radiophosphorus emits a beta particle with a much higher energy than does radiocarbon, one can place two sheets of X-ray film on the developed chromatogram, with the result that the film next to the paper will be exposed by the beta particles from both the phosphorus and carbon while the top film will be exposed by radiation from the phosphorus only, the carbon betas having been entirely absorbed by the first film. Then if one waits until a number of half lives of P^{32} (14 days) have expired and places a fresh film on the chromatogram, a radioautograph of the carbon only is obtained. By superimposing the two films we can see at once which compounds have become labeled with both tracers and this in turn indicates which carbon compounds are phosphorylated.

If an experiment is carried out in which the plants are exposed to radiocarbon and radiophosphorus sufficiently long to saturate the soluble compounds in the plant with both tracers and if the usual chromatographic analysis is carried out, one can then count the carbon activity and phosphorus activity in a known compound, using suitable absorbers. By comparing this ratio (P^{32}/C^{14}) with the known ratio of P^{31}/C^{12} for phosphoglyceric acid, Benson (*25*) was able to calculate the P^{31}/C^{12} ratio for other unknown compounds appearing on the chromatogram by measuring the P^{32}/C^{14} ratio for these compounds.

The use of tracer phosphorus is also applicable to phosphate compounds which are not labeled by carbon during photosynthesis. Several studies with radiophosphorus and photosynthetic organisms have been performed. In most cases the fractionation of the products has been accomplished by means of extractions. Aronoff and Calvin (*26*) found no direct connection between gross formations of organic phosphorus compounds and photosynthesis or photochemical reductions when they exposed *Chlorella* to a solution of radiophosphate in the light and in the dark. Kamen (*27*) and Simonis and Grube (*28*), working with various organisms and conditions of exposure and extraction,

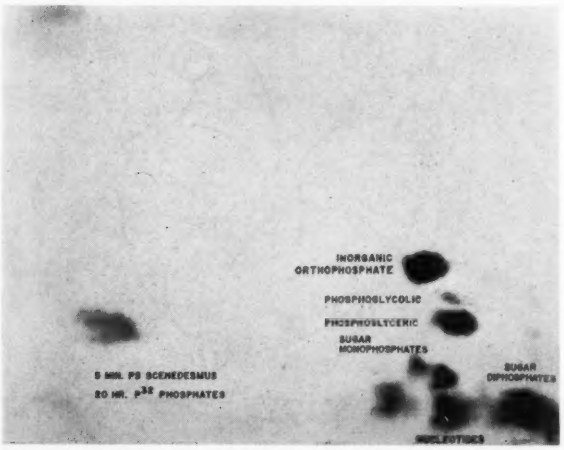

Figure 14. P^{32}-Labeled Compounds Found in the Same Experiment as That Described in Figure 13

This radiogram was obtained by exposing a sheet of X-ray film to the same chromatogram as in Figure 13 shortly after the experiment was performed and using a sheet of exposed film to filter out the low-energy C^{14} beta particles.

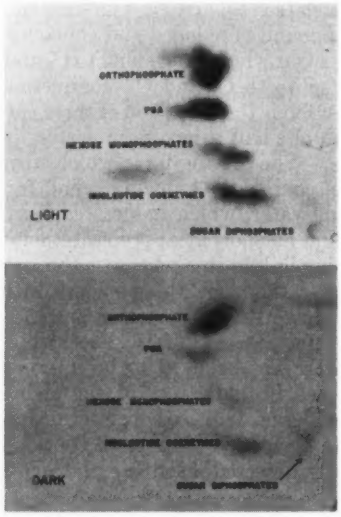

Figure 15. Radiogram of P^{32}-Labeled Compounds Formed by 1 Minute Exposure of *Scenedesmus* to P^{32}-Labeled Phosphate in the Light and in the Dark

In each case, the *Scenedesmus* had been adapted to photosynthesis in the light previous to the experiment. The exposures in the two radiograms shown cannot be compared as to intensity, due to differences in experimental conditions and film exposure times.

found some differences between the P^{32} content of certain fractions after exposure to tracer phosphorus in the light and that of these fractions in the dark.

Just as in the case of the studies with radiocarbon, a much more satisfactory method of studying the labeling of compounds with tracer phosphorus during photosynthesis in the light and in the dark is to be found in the use of chromatography and radioautography as methods of analysis and detection. Such experiments are now underway in the Berkeley laboratory[3] and only preliminary results can be reported at this time. In Figure 15 we see the radioautographs of the products of one-minute fixation by *Scenedesmus* of P^{32} in the dark and in the light. The intensities of the spots are not comparable in these experiments, but the rapid conversion of inorganic to organic phosphate is worth noting. In the one-minute photosynthesis experiment 70 per cent of the soluble compounds is in the form of organic phosphates. In this experiment the algae were washed three times with distilled water before killing (requiring about 15 seconds) and then killed and extracted with 80 per cent ethanol in water, 20 per cent ethanol in water, and water. The combined extracts contained 80 per cent of the total fixed activity. It is hoped that through such experiments the path of phosphorus in photosynthesis may be traced.

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[3] Private communication from M. Goodman and D. Bradley.

EDITOR'S OUTLOOK

(*Continued from page 273*)

1. Recognition of the fact of constant change in all things —of the process nature of life and the universe.
2. Recognition of the uniqueness of all things—atoms, stars, persons, places, events, elements, etc.
3. Drawing of inferences, hypotheses, theories, etc., only after careful observation of the behavior of things.
4. Use of inferences, theories, etc., only as long as they offer the best explanation of available data.
5. Discarding or revising of inferences, theories, etc., as soon as they no longer adequately explain available data.
6. Refusal to regard a "principle" or a "classification" or a "category" as anything more than a man-made device for convenience in comprehending and communicating and testing.
7. Realization that theories, principles, etc., exist only as abstractions whose chief function is not to provide dogma to be taught but rather questions to be answered by further research.
8. Eagerness to accept tested truth no matter what its origin.
9. Willingness and ability to announce as plainly as possible (in the time-honored manner of mathematicians) any assumptions, including definitions of basic terms, involved in reasoning.
10. Awareness of the difference between facts and judgments and determination never for any reason to willfully confuse the two in reporting.
11. Understanding that there is always another word to be said, another inference to be drawn, another principle to be discovered.

Many more such habits could be listed but these should be enough to permit a few generalizations about the kind of personality and the nature of the moral and ethical standards they would tend to foster. Would not such habits of mind in an individual *mean* that he would be honest, truthful, cautious, dependable, openminded, humble, fair, reasonable, impartial, democratic, just? Would not such habits of mind in a teacher tend to promote such habits in his students? Would not such promotion be character training and moral and ethical teaching at their best?

ASPECTS OF ISOMERISM AND MESOMERISM

II. Structural Isomerism

RICHARD L. BENT
Eastman Kodak Company, Rochester, New York

IT MUST be admitted that there is still a certain amount of confused thinking concerning isomerism (particularly stereoisomerism), and that the precise relationships that exist among the different types are sometimes misunderstood. In some respects, the whole field of isomerism resembles a complicated jigsaw puzzle. Like the latter, one can start anywhere and end anywhere (or very nearly so), but not until a considerable number of parts are in their proper places will the general pattern be discernible.

Unfortunately, the nomenclature in isomerism is not yet completely standardized. Thus, certain terms often vary slightly in meaning from text to text. Considerable care has been taken to see that the particular definitions adopted in the present series of papers are consistent with one another and are not self-contradictory.

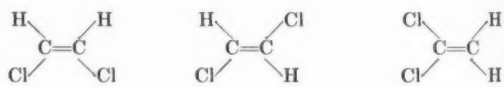
Although the present paper is concerned primarily with structural isomerism, it is difficult to avoid using a few stereochemical terms. To avoid repetition, such terms are only briefly discussed here; a more thorough explanation will be given in Part III. However, a complete chart of isomerism is included in this issue. The chart would lose much of its usefulness if it did not cover all branches of isomerism.

Let us first consider the isomerism that occurs in the *covalent* compounds. When two or more compounds are found to possess the *same molecular formula*,[1] they are called *isomers* of one another, and the phenomenon is termed *isomerism*. Since isomeric molecules contain the same number of atoms of each element—by definition—the different properties exhibited by the various isomers can only be explained by postulating differences in the arrangements of the component atoms. The word *arrangement* as used here, should be carefully noted, for it can mean either *structural* or *configurational* arrangements.

Many cases of isomerism can be explained solely on the basis that the respective molecules differ in regard to which atom is linked to which, no consideration being given to the spatial or configurational aspects of the molecules. For example, the arrangement of atoms in a particular molecule might be such that A is linked to B, and B to C, while the arrangement in another molecule might be A to C, and C to B. In other words, the two molecules possess identical molecular formulas but different structural formulas (A—B—C and A—C—B, respectively). This type of isomerism is called *structural isomerism*.

Let us consider briefly the configurational arrangements of atoms. Different compounds exist which not only possess identical molecular formulas but also identical structural formulas, indicating that an even more subtle type of isomerism can occur. Compounds of this type are called *stereoisomers* of one another, and the phenomenon is termed *stereoisomerism*. The differences between molecules which contain the same number of atoms of each element, and the same structural arrangements of those atoms, can only be explained on the basis of different spatial or configurational arrangements.

When a compound is said to be an isomer of another, it must be either a structural isomer or a stereoisomer—it cannot be both at the same time. It is perfectly possible, however, for a compound to be structurally isomeric with a second compound and spatially isomeric (stereoisomeric) with a third. For example, *cis*-dichloroethylene, I*a*, is spatially isomeric to *trans*-dichloroethylene, I*b*, but structurally isomeric to



(I*a*) (I*b*) (I*c*)

unsym-dichloroethylene, I*c*. The three formulas are to be regarded as *configurational* formulas and they portray the relative spatial positions of both the atoms and the bonds in the respective molecules. Compounds I*a* and I*b* possess the same structural formula, $CHCl{=}CHCl$. Compound I*c*, on the other hand, possesses a *different* structural formula, $CCl_2{=}CH_2$ (all three, of course, possess different configurational formulas).

It should be pointed out that if the covalent bond did not exist, no isomerism of any type would be possible. A compound whose atoms are held together solely by ionic linkages is not capable of exhibiting isomerism, although, of course, it might exist in one or more crystalline forms (*i. e.*, exhibit polymorphism). Isomerism can only occur in a compound if the following conditions are satisfied: (1) the compound must consist either of neutral molecules (whose atoms are, of course, covalently linked), or of ions, at least one of which must consist of a group of covalently linked atoms; (2) the

[1] See Part I for a discussion of the different types of formulas.

atoms in the molecule, or ion, must be capable of existing in more than one structural or spatial arrangement.

Because the majority of carbon compounds contain covalent rather than ionic linkages, the usual definitions of the different types of isomerism in organic chemistry (and those in the present paper) are based on the concept of the covalent molecule, *i. e.*, they are based on the concept of two or more compounds possessing identical *molecular* formulas.[2]

Unfortunately, in regard to the ionic compounds there is little agreement as to the basic definitions; in fact, some writers even object to the use of the term isomerism in connection with such compounds. However, the present writer believes the following simple definition to be quite satisfactory: When two or more ionic compounds are found to possess the *same ionic formula* (see Part I), they can be said to exhibit isomerism.

Propyl alcohol, $CH_3CH_2CH_2OH$, and isopropyl alcohol, $CH_3CHOHCH_3$, for example, possessing the same *molecular* formula, C_3H_8O, are isomeric because their respective *molecules* contain different structural arrangements of the atoms. The sodium derivatives of the two alcohols, sodium propoxide, $[CH_3CH_2CH_2O]^-[Na]^+$, and sodium isopropoxide, $[CH_3CHOCH_3]^-[Na]^+$, possessing the same *ionic* formula, $[C_3H_7O]^-[Na]^+$, on the other hand, can be called isomeric[3] because their respective *ion-pairs* contain a group of atoms possessing different structural arrangements in the two compounds.[4]

Structural isomerism is subdivided into smaller classes such as chain isomerism, position isomerism, tautomerism, etc. *Stereoisomerism* is divided into two main classes, optical and geometric isomerism, which are subdivided into smaller classes.

For some time the author has felt the need of a chart which would show *in detail* the relationships that exist between each type of isomerism (in organic chemistry). An attempt to construct such a chart was made and it is presented in this issue. As complete a description as possible of each type is given under the appropriate heading, for it was felt that this would materially add to its usefulness. Certain obsolete terms, such as pseudomerism and cryptomerism, are omitted (although such terms are explained later), for these terms are superfluous today. In order to avoid unnecessarily complicated statements, the definitions throughout are restricted to covalent molecules. The chart should be consulted carefully before examining the specific examples to follow, because the explanations given there are not repeated in the text.

SAME CHEMICAL TYPES

Chain Isomerism

(II*a*) *n*-Butane (II*b*) iso-Butane

(III*a*) Bicyclo [4.2.0] octane (III*b*) Bicyclo[3.3.0]octane

This type of isomerism is also called *nuclear* isomerism, although the term *chain* isomerism is to be preferred. Apart from the fact that nuclear isomerism refers to a special phenomenon in nuclear physics, the use of the term "nucleus" is unnecessary. Thus, Compounds II*a* and II*b* are said to exhibit chain isomerism because of the different ways in which the carbon atoms are linked up. Since such atoms can be linked up in rings as well as in chains, compounds such as III*a* and III*b* can logically be called chain isomers.

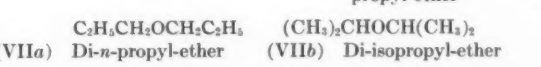
Position or Substitution Isomerism

(IV*a*) 1-Butene (IV*b*) 2-Butene

(V*a*) α-Chloronaphthalene (V*b*) β-Chloronaphthalene

Metamerism

(VI*a*) Diethylether (VI*b*) Methyl-*n*-propyl-ether



(VII*a*) Di-*n*-propyl-ether (VII*b*) Di-isopropyl-ether

This type of isomerism occurs with the ethers, amines, esters, etc. The term *metamerism* is seldom used today probably because chain and position isomerism together really cover this class of isomerism, if there usual meaning is extended slightly. Thus, in example VI, the two isomers differ only in the position of the oxygen in the chain, just as 1-butene, IV*a*, and 2-butene, IV*b*, differ only in the position of the double bond in the same fundamental chain. It would thus be perfectly logical to classify this pair of "metamers" as position or substitution isomers. In example VII, the

[2] A covalent bond may, of course, have a large amount of ionic character associated with it, *e. g.*, a carbon-fluorine bond. Nevertheless, such a bond retains the important properties of a covalent bond (electrons are still being shared; the bond has directional properties, etc.).

[3] Compounds such as methyl ammonium formate, $[CH_3NH_3]^+[HCOO]^-$, and ammonium acetate, $[NH_4]^+[CH_3COO]^-$, where both the anions and cations are different, have been called *stoichiomers* by some writers; see WHELAND, G. H., "Advanced Organic Chemistry," 2nd ed., John Wiley & Sons, Inc., New York, **1949**, p. 34.

[4] The negative charges on the anions, of course, have no effect on the structural arrangements of atoms in the groups. Because the different structural arrangements occur in the anion, in the above example, and because each of the ion pairs does not normally possess an independent existence (in contrast to neutral molecules), some writers feel that the isomerism should be said to occur in the anion rather than in the compound. Nevertheless, an ionic compound is represented by a complete ionic formula and not by just one ion. For this reason, the writer feels that any definition of isomerism should refer to the compound as a whole and not just to one part of that compound.

ISOMERISM

When two or more compounds are found to possess the same molecular formula they are said to exhibit isomerism. (The molecular formula tells us how many atoms of each element are present in a molecule of the substance.) Each isomer possesses an identical number of atoms of each element and yet the individual isomers are chemically and physically distinct from one another (with the exception of enantiomorphic isomers which only differ in regard to one important physical property, viz., their opposite rotatory effect on polarized light). For isomerism to be possible between such molecules, each must possess a different arrangement of the constituent atoms. The two broad classifications of isomerism that follow are dependent on the particular nature of these arrangements. Molecules exhibiting structural isomerism differ in regard to which atom is linked to which. Molecules exhibiting stereoisomerism do not differ in this regard, but differ only in the spatial arrangements of the atoms.

STRUCTURAL ISOMERISM

When two or more compounds are found to possess the same molecular formula but different structural formulas they are said to exhibit structural isomerism. Structural isomers differ in regard to which atom is linked to which. (The structural formula tells us how many atoms of each element are present in a molecule of the substance, which atom is linked to which, and in each case whether the linkage is single, double or triple.[1] A structural formula does not, however, tell us anything about the spatial relationships of the atoms; such information is contained in a configurational formula.)

Type of structural isomerism shown by compounds of the same chemical type.

CHAIN OR NUCLEAR ISOMERISM

Irrespective of the chemical nature of the compound in general there are different ways of linking up the carbon atoms (and other atoms to a certain extent) giving rise to a number of distinct structural arrangements, thus for example, the fundamental chain of carbon atoms might be branched or it might consist of one "straight" chain only. Irrespective of whether any other types of isomerism are also present at the same time, those isomers which differ in the fundamental chain arrangements would be examples of chain or nuclear isomerism.

POSITION OR SUBSTITUTION ISOMERISM

These isomers differ in the position occupied by an atom, group or function (such as a double bond) with reference to the same fundamental carbon chain (or ring).

METAMERISM

When different radicals (of the same chemical type) are attached to the same polyvalent element and yet give rise to compounds possessing an identical molecular formula, they are said to possess this type of isomerism (e.g., diethylamine and methyl propylamine). Almost an archaic term.

PROTOTROPY

This refers to tautomeric systems which are the result of a mobile hydrogen ion. By far the most common type. Various types which could be classified as follows:

(a) Dyad
(b) Triad
(c) Ring-chain
(d) Trans-annular

ANIONOTROPY

This refers to tautomeric systems where the migrating group is a negative ion (such as the hydroxyl) rather than the hydrogen ion.

FUNCTIONAL GROUP ISOMERISM

Type of structural isomerism shown by compounds of distinct chemical types, can be divided into two classes.

(a) Exhibited by compounds which, although they belong to a different chemical class, are not directly interconvertible (e.g., ethyl alcohol and dimethyl ether).

(b) TAUTOMERISM OR DYNAMIC ISOMERISM

This constitutes, in general, a special case of functional group isomerism which can be termed "dynamic." This is said to occur when two or more structural isomers are mutually interconvertible under normal conditions, thus forming a mixture which is in dynamic equilibrium. In general, the isomers involved are of different chemical types (e.g., keto-enol, etc.) but some cases of structural dynamic isomerism involve two or more compounds of the same chemical type, and are therefore examples of dynamic position or substitution isomerism (e.g., the pyrazole tautomerism).

GEOMETRIC TAUTOMERISM[2]

Strictly speaking, a branch of tautomerism involving geometric isomers only; thus, for example, the chair and boat forms of cyclohexane form a dynamic equilibrium mixture.

MUTAROTATION

A special case of dynamic isomerism involving either (a) the tautomeric transformation of one optical isomer into another or (b) the tautomeric transformation of one structural isomer (possessing at the same time one or more asymmetric centers) into another, the latter now possessing different asymmetric centers due to the change in structure. With both types the optical rotatory power of a freshly prepared solution of the compound will change under a variety of conditions, until an equilibrium value is set up which will not be zero (unlike racemization).

RACEMIZATION

The transformation of one-half of the molecules of an optically active compound into molecules which possess exactly the opposite configuration; i.e., the transformation of one-half of one enantiomorph into the other. The resultant equimolecular (racemic) mixture is said to be externally compensated, for the lack of rotary power is due to a statistical balance between equal numbers of dextro- and laevo-rotatory molecules (as in mutarotation a dynamic equilibrium is involved).

[1] Because a covalent bond is now known to consist of a shared pair of electrons, the classical definition of a structural formula (given above) has been extended somewhat. Nevertheless, if two or more structures differ only in the position of electrons, rather than atoms, in the great majority of cases they are not considered to be separate isomers but rather to possess a single (mesomeric) electronic structure (for an important exception to this generalization see discussion of tautomerism in the text).

[2] This branch of tautomerism should have a connecting line linking it to Geometric Isomerism on the opposite page.

STEREOISOMERISM

When two or more compounds are found to possess the same molecular formula and the same structural formula but different spatial or configurational formulas, they are said to exhibit stereoisomerism. Stereoisomeric molecules differ in the spatial relationships of the atoms but do not differ in regard to which atom is linked to which. (A configurational formula tells us how many atoms of each element are present in a molecule of the substance, which atom is linked to which, the number and type of linkages involved, and the relative spatial positions of the atoms.) Although stereoisomerism is sometimes called three-dimensional isomerism, the term spatial isomerism is to be preferred, because stereoisomerism can also be exhibited by completely planar molecules (e.g., cis and trans dichloroethylene). Similarly, the term spatial formula is preferable to the term three-dimensional formula, because such formulas can also represent planar, or even linear, molecules.

OPTICAL ISOMERISM[1]

Optical isomerism is said to occur between two stereoisomers if they exhibit any of the following effects on polarized light:

(a) The isomers rotate the plane of polarization to a different degree in either the same direction or in opposite directions.
(b) They rotate the plane to the same degree in opposite directions.
(c) They have no effect on the plane because of so-called "internal" compensation (see diastereoisomerism).

GEOMETRIC ISOMERISM

This branch of stereoisomerism is difficult to define.[3] It is usually described (loosely) as that type of stereoisomerism which is dependent upon some form of restricted rotation,[4] enabling the component parts of the molecule to occupy different spatial positions. The various classifications of compounds possessing restricted rotation can be listed as follows:[5]

(a) Ethylenic or double-bond type.
(b) Due to the rigidity of the ring structure in (1), certain monocyclic compounds and (2) certain fused cyclic compounds.
(c) Single-bond, steric-hindrance type, as exhibited in certain terphenyl compounds.

ENANTIOMORPHISM

This includes those optical isomers which are in class (b). Any two isomers are enantiomorphic only if there is a nonsuperimposable, mirror-image relationship existing at all times between their respective molecules. The following types of molecules (whether freedom of rotation is present in the molecule or not) will always possess a nonsuperimposable mirror image and will therefore be capable of giving rise to enantiomorphic isomers.

Those molecules which possess an individual asymmetric[2] carbon atom.

For classification purposes we can divide this group into the following types:
(1) Acyclic or straight-chain compounds possessing one asymmetric carbon atom.
(2) Monocyclic compounds of the same category.
(3) Fused cyclic compounds of similar category.

If the molecule possesses n different asymmetric carbon atoms, then a total of 2^n optical isomers, or $2^n/2$ enantiomorphic pairs, will be possible (except in certain fused cyclic compounds where the number will be less for steric reasons).

If the molecule possesses one or more similar asymmetric carbon atoms, then certain isomers, the meso-compounds, are capable of existence which do not possess an enantiomorphic partner. Thus, the total number of isomers and enantiomorphic pairs possible will be less in such cases than if the asymmetric carbon atoms were all different.

Those molecules which are asymmetric[2] as a whole and yet do not possess an individual asymmetric carbon atom.

Many different types which can be listed as follows:
(a) Allene type.
(b) Spirane type.
(c) Inositol type.
(d) Steric hindrance type:
  (1) Biphenyl type.
  (2) Naphthalene and benzene type.

DIASTEREOISOMERISM

This includes optical isomers of types (a) and (c). Any two optical isomers are said to be diastereoisomers of one another if their respective molecules do not at any time exhibit a mirror-image (enantiomorphic) relationship to one another. Diastereoisomers possess different physical and chemical properties, in contrast to enantiomorphs which possess identical properties (with one important exception in regard to their effect on polarized light).

Type (a): A molecule containing two different asymmetric carbon atoms, for example, is capable of existing in four optically active forms (i.e., two pairs of enantiomorphs). The relationship between any one of these isomeric molecules and its mirror image is said to be enantiomorphic. The relationship between any one isomer and either of the isomers constituting the second pair of enantiomorphs is said to be diastereoisomeric.

Type (c): Meso-compounds: Only those compounds that possess two or more similar asymmetric carbon atoms are capable of possessing meso-isomers. Tartaric acid, for example, containing two such atoms, exists in three different stereoisomeric forms. Two of these exhibit an enantiomorphic relationship to one another. The third (the meso-isomer) exhibits a diastereoisomeric relationship to the other two. As explained in Part III, an appreciable number of molecules of a meso-isomer (of the tartaric acid type) possess a zero over-all optical activity, not, however, by "internal" compensation (as commonly described), but actually by "external" compensation (while it is true, at any particular instant, that a few molecules are internally compensated, the great majority are externally compensated).[6]

In addition to the two classes listed above, there is another type of diastereoisomerism (so-called) that is exhibited by certain "organic" salts, i.e., salts formed from an organic acid and an organic base. If a dl-acid (i.e., an equimolecular mixture of the dextro and laevo forms of the acid) is neutralized with a d-base, for example, we obtain a mixture of the dd-salt and the dl-salt. Although the d and l forms of the acid have identical properties (except for opposite rotatory power), the properties of the dd and dl salts are not identical, and the two salts can therefore be separated. Salts of this type are called diastereoisomers.

ISOMERIZATION

The process whereby any isomer, whether structural, geometric, or optical, is converted into another, usually requiring special conditions of temperature, pressure or catalysts.

[1] In the above chart all statements concerning the optical activity of an isomer refer to an appreciable number of molecules of the isomer, i.e., they refer to an average, or statistical optical activity. As explained in Part III, if a molecule does not at any time (if freedom of rotation is present) possess a plane or point of symmetry (or, in theory, an alternating axis), i.e., if it is asymmetric[2] at all times, it will possess optical activity of the same sign, though to a different degree, in all possible spatial positions. The statistical optical activity of an appreciable number of such molecules will, of course, also possess optical activity of the same sign. Similar reasoning applies to the mirror image, or enantiomorph, of the above molecule (in its various spatial forms) except that it will always exert an opposite rotatory effect on polarized light. If, however, a molecule is capable at any time of possessing one of the above elements of symmetry, the statistical optical activity is always found to be zero.[6] In such cases the optical activity of an individual molecule varies, in general, both in sign and in magnitude. Since, however, the probability of such a molecule possessing a particular degree of rotatory power in one direction is always equal to the probability of it possessing the same rotatory power in the opposite direction (because of the symmetry of the molecule), the individual activities of the molecules cancel out (by external compensation).

[2] When describing a molecule, the term asymmetric is commonly used instead of the more accurate term disymmetric. Strictly speaking, the word asymmetric implies the absence of all elements of symmetry (the plane, point and alternating axis, as well as the unimportant ordinary axis). Because it is possible for an optically active molecule to possess an ordinary axis of symmetry, however, it is more accurate to call it disymmetric. Nevertheless, it is still perfectly correct to speak of an asymmetric carbon atom (see Part III).

[3] See discussion in Part III; see also Wheland, G.W., "Advanced Organic Chemistry," John Wiley and Sons, Inc., New York, 1949, pages 194 and 217.

[4] Although this restriction of rotation does occur in the saturated cyclic compounds, it is not a strictly necessary condition in order that the isomerism may occur (see reference 3).

[5] See Sshriner, R. L., et al, in Gilman's "Organic Chemistry," Vol. I, 2nd ed., John Wiley and Sons, Inc., New York, 1943, pages 444-487.

[6] See Meller, C. R., THIS JOURNAL, 24, 900 (1947); and Mowery, D. F., THIS JOURNAL, 29, 138 (1952); and also Wheland, G. W., reference 3, pages 144 and 191.

oxygen atom can be considered as being fixed (in the sense that both molecules are symmetrical), while the connecting groups differ in their chain structure. Therefore, this pair of isomers could legitimately be classified under chain or nuclear isomerism.[5]

DIFFERENT CHEMICAL TYPES

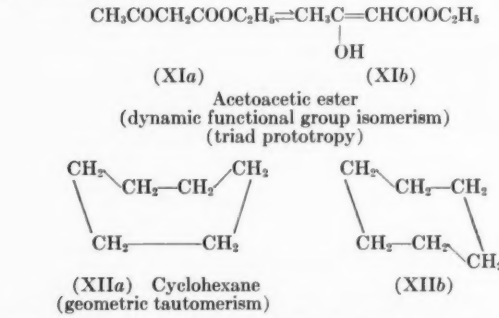
Functional Group Isomerism

$ClCH_2CH_2COOH$	CH_3OCH_2COCl	$HOCH_2COCH_2Cl$
(VIII) β-Chloro-propionic acid	(IX) Methoxy-acetylchloride	(X) α-Chloro-α′-hydroxy-acetone

The isomers mentioned in the previous section have very similar properties, owing to the identical chemical nature of the substances involved. Functional group isomers, on the other hand, possess very different chemical and physical properties, for, by definition, members of this group possess different "active" or "functional" groups.

Tautomerism or Dynamic Isomerism

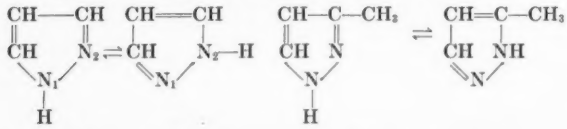
Although tautomerism was at one time exclusively concerned with prototropy, it now includes such phenomena as anionotropy, geometric tautomerism, etc. (see chart). By definition, tautomeric substances possess two or more molecular forms which are mutually interconvertible (under normal conditions). The term *molecular* form is used, rather than *isomeric* form, since it is possible for the tautomers to be identical, as in pyrazole, XIII.



(XI*a*) (XI*b*)

Acetoacetic ester
(dynamic functional group isomerism)
(triad prototropy)

(XII*a*) Cyclohexane (XII*b*)
(geometric tautomerism)



(XIII*a*) (XIII*b*) (XIV*a*) (XIV*b*)

Tautomeric forms of pyrazole (identical) (dyad prototropy)

Tautomeric forms of methyl pyrazole (not identical) (dynamic position or substitution isomerism—dyad prototropy)

Tautomeric substances can be divided into three main classes: (1) those substances whose molecules possess more than one *structural* formula, *e. g.*, acetoacetic ester, XI; methyl pyrazole, XIV; (2) those substances whose molecules possess more than one *configurational* formula, *e. g.*, cyclohexane, XII; glucose, XVI; (3) those substances whose molecules possess *identical* structural and configurational formulas, *e. g.*, pyrazole, XIII. Because of the existence of class (3), strictly speaking, all tautomeric substances are not examples of dynamic isomerism, since identical molecules (identical in the sense of their properties) obviously cannot be called isomeric. In only one sense can the two forms of pyrazole not be considered identical. In the form, XIII*a*, the tautomeric hydrogen belongs to N_1, while in the form, XIII*b*, it belongs to N_2 (the numbers would normally be reversed in the form, XIII*b*). In common practice, the terms tautomerism and dynamic isomerism are used interchangeably and for this reason the chart was not made unnecessarily complicated.

Occassionally it is possible to isolate, or prepare, one (or more) of the tautomeric forms in a pure or relatively pure state, and these separable forms are sometimes called desmotropes. Sooner or later, however, at normal temperatures, the presence of impurities or catalysts, etc., will cause the pure species to change into another form (or forms) until equilibrium is again established. The time required to reach equilibrium can vary within wide limits—anywhere from a fraction of a second to days or months. How rapidly a particular tautomeric change occurs will depend upon the nature of the tautomer, its purity, the temperature, the presence of catalysts, etc. The great majority of tautomeric systems result from the shifting of an atom or group (generally hydrogen) from one part of the molecule to another.

Prototropy. Tautomeric systems which involve a mobile hydrogen (in ionic form) are said to exhibit prototropy. The great majority of tautomers belong to the prototropic class.

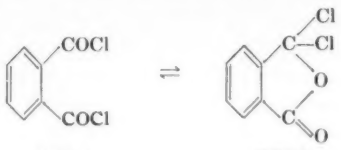
Dyad Prototropy. In this class of tautomers the hydrogen can be considered as moving from one atom to another which is adjacent to it, *e. g.*, pyrazole, hydrogen cyanide, etc. This does not mean, of course that the hydrogen atom which enters the molecule in a new position is necessarily the same hydrogen that left the molecule (the entering hydrogen might be from an adjacent molecule, for example, or from the solvent).

Triad Prototropy. If, in a chain of three atoms, a mobile hydrogen can be considered as moving from the first atom to the third, it is said to exhibit triad prototropy. A very large number of tautomers belong to the triad class, which includes such types as keto-enol, nitroso-oxime, amido-imido, three-carbon systems, etc.

Ring-Chain Tautomerism. When a substance can act as if it possessed both a cyclic structure and an open-chain structure, it is said to exhibit ring-chain tautomerism. In the case of phthaloyl chloride, XV (which

[5] There is not complete agreement in the standard texts as to exactly what types of compounds should or should not be called chain or position isomers, and therefore definitions differ somewhat. However, because the differences between chain isomerism, metamerism, and position isomerism are not very sharp, it would really be more satisfactory to classify all three types under one heading. In each case, the isomers are of the same chemical type and differ in regard to which atom is linked to which.

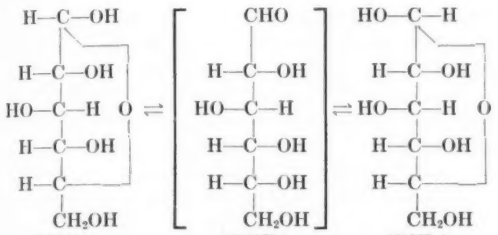
exhibits *anionotropic* tautomerism), the molecule is capable of existing in one cyclic form and one



(XV*a*) (XV*b*)

Phthaloyl chloride

open-chain form. However, with glucose, XVI, two ring forms are possible (*i. e.*, two *six*-membered rings) in addition to the intermediate open-chain aldehyde form.



(XVI*a*) (XVI*b*) (XVI*c*)

α-d-Glucose Aldehyde form *β-d*-Glucose

Anionotropy. Tautomeric systems which involve a mobile anion are said to exhibit anionotropy. The anions OH^-, Cl^-, and CH_3COO^-, for example, can, under certain conditions, undergo this type of tautomerism. The allyl derivatives frequently exhibit anionotropy, as represented by the general formula XVII, X being the mobile group. As regards their mobility or ease of transfer the following order generally holds: Cl>OAc>OH. This type of tautomeric change is much less common than prototropy. It is not to be assumed, of course, that all mobile groups (other than hydrogen) are necessarily anionic.

(XVII) $RCH(X)CH{=}CHR \rightleftharpoons RCH{=}CHCH(X)R$

OBSOLETE TERMS

The word isomerism was first used by Berzelius in 1831 to describe those substances which possess the same percentage composition but had different properties. He divided the substances exhibiting isomerism into two groups. The term *polymerism* was used to describe those compounds which possessed the same percentage composition but had different molecular weights, and the term *metamerism* to describe those compounds which possessed the same molecular formula but had different properties. What Berzelius called metamerism is now called isomerism.

As noted previously, the term metamerism is now restricted to a special class of structural isomers and is nearly obsolete. The phenomenon of polymerism (although the actual word "polymerism" is seldom used) by definition now has no connection with isomerism. A compound that is a polymer of another (whether theoretical or practical) is not regarded as being isomeric because isomerism is restricted solely to compounds possessing either identical molecular or ionic formulas.

The term *pseudomerism* (now obsolete) was first used by Laar, on the basis of Von Baeyer's researches, in 1885 to describe what is now called *tautomerism.* The prefix "pseudo" was used because he believed that the isomers involved in the dynamic equilibrium were not capable of independent existence.

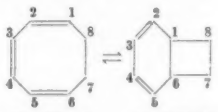
Jacobsen in 1887 used the term *desmotropism* (meaning literally "bond-turning") to describe the phenomenon, since all examples of tautomerism which were then known involved a rearrangement of bonds (in addition to a rearrangement of atoms). The term desmotropism is no longer used, although the term desmotrope is occasionally used to describe a tautomer (*i. e.*, one of the tautomeric forms) which can be prepared in a pure state. It seems likely that this term also will become obsolete.

Another archaic term is *cryptomerism* which referred to those tautomeric systems where it was not known which isomer was present in the greatest concentration.

When a compound could apparently be represented by two or more structural formulas differing only in the arrangement of bonds, *i. e.*, electrons, they were at one time called *valency* isomers and at a later date *electronic* isomers. To a large extent these terms have fallen into disuse, because in the majority of cases the phenomenon involved is actually mesomerism, or reasonance, and not tautomerism. Thus, as explained in detail in Part I, benzene is not considered to consist of two separate isomeric forms in dynamic equilibrium; the actual electronic state of the molecule is considered to be intermediate between the two extremes.

Nevertheless, there are a few exceptions to the above generalization. Certain compounds are capable of exhibiting both ring closure and ring opening, involving changes in "bond structure" only, that can definitely be classified as examples of valency tautomerism rather than mesomerism.

Thus, 1,3,5-cyclo-octatriene, XVIII*a*, is found to be in dynamic equilibrium with bicyclo [4.2.0] octa-2,4-diene, XVIII*b* (on heating).[6]



(XVIII*a*) (XVIII*b*)

The electronic changes that occur involve the interchange of single and double bonds. However, because of the large change in the distance between atoms 1 and 6 which occurs when a bond is formed between the two

[6] See Cope, A. C., *et al.*, "Cyclic polyolefins. Valency tautomerism," in the abstracts of papers of the Division of Organic Chemistry, American Chemical Society, presented at Buffalo, New York, March 1952, p. 6k. See also Cope, A. C., *et. al.*, *J. Am. Chem. Soc.*, **74**, 4867 (1952).

atoms, it would appear that mesomerism is ruled out.[7] If, however, the distances between atoms 1 and 6 in the two compounds were approximately the same *at all times*, then undoubtedly, the phenomenon of mesomerism only would be involved.

[7] See WHELAND, G. W., "The Theory of Resonance," John Wiley & Sons, Inc., New York, **1944**, p. 6.

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THE ACTION OF NITRIC ACID ON METALS

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A GENERAL equation for the action of nitric acid on metals has been computed.

The products formed by the action of metals on nitric acid are: (1) the nitrate of the metal, (2) water, and (3) one or more reduction products (nitrous oxide, nitric oxide, nitrogen trioxide, nitrogen peroxide, nitrous acid, hyponitrous acid, nitrogen, ammonia, hydroxylamine). According to Mellor,[2] anyone of the foregoing reaction products may be obtained in the reaction, dependent upon the conditions and the metal used.

All the above reactions of nitric acid on the metals can be represented by a single equation:

$$2\frac{n\mathrm{M}}{v} + 2(n+1)\mathrm{HNO_3} \rightarrow 2\frac{n\mathrm{M}}{v}(\mathrm{NO_3})_v + (n+1)\mathrm{H_2O} + \mathrm{N_2O}_x \quad (1)$$

where M is the metal, v the valency of that metal, and n, x are any two integers (positive, negative, or zero) related by the equation $5 - n = x$.

In cases of monovalent and divalent metals the equation (1) is reduced to simple forms by substituting $v = 1$ and 2, thus:

$$2n\mathrm{M} + 2(n+1)\mathrm{HNO_3} \rightarrow 2n\mathrm{M(NO_3)} + (n+1)\mathrm{H_2O} + \mathrm{N_2O}_x \quad (2)$$

$$n\mathrm{M} + 2(n+1)\mathrm{HNO_3} \rightarrow n\mathrm{M(NO_3)_2} + (n+1)\mathrm{H_2O} + \mathrm{N_2O}_x \quad (3)$$

Similarly, equations for higher valent metals can be deduced.

INTERPRETATIONS

(1) When nitrous oxide, nitric oxide, nitrogen trioxide, and nitrogen peroxide are produced. Here x is always a positive integer and is equal to the number of oxygen atoms in a molecule of the oxide of nitrogen formed, written in the form of N_2O_x. Thus, substituting $x = 1$, 2, 3, or 4 in equation (1) gives the following equations for the action of nitric acid on divalen tmetals:

$$4\mathrm{M} + 10\mathrm{HNO_3} \rightarrow 4\mathrm{M(NO_3)_2} + 5\mathrm{H_2O} + \mathrm{N_2O}$$
$$3\mathrm{M} + 8\mathrm{HNO_3} \rightarrow 3\mathrm{M(NO_3)_2} + 4\mathrm{H_2O} + \mathrm{N_2O_2}$$
$$2\mathrm{M} + 6\mathrm{HNO_3} \rightarrow 2\mathrm{M(NO_3)_2} + 3\mathrm{H_2O} + \mathrm{N_2O_3}$$
$$\mathrm{M} + 4\mathrm{HNO_3} \rightarrow \mathrm{M(NO_3)_2} + 2\mathrm{H_2O} + \mathrm{N_2O_4}$$

(2) When nitrous and hyponitrous acids are produced. These acids are produced as a secondary reaction between the corresponding acid-anhydrides (oxides of nitrogen formed) and water formed in the reaction; hence the equations when these acids are produced by the action of nitric acid on metals can be deduced by substituting values of x corresponding to the acid-anhydrides.

(3) When nitrogen is one of the products. In this case the value of x is taken as zero, so that N_2O_x becomes N_2. The equation, for a divalent metal, is as follows:

$$5\mathrm{M} + 12\mathrm{HNO_3} \rightarrow 5\mathrm{M(NO_3)_2} + 6\mathrm{H_2O} + \mathrm{N_2}$$

(4) When ammonia is one of the products. N_2O_x is equivalent to N_2H_{-2x} plus xH_2O. Hence, to obtain ammonia, the value of x is -3, giving the following equation (for a divalent metal):

$$8\mathrm{M} + 18\mathrm{HNO_3} \rightarrow 8\mathrm{M(NO_3)_2} + (9\mathrm{H_2O} + \mathrm{N_2O_{-3}}) \rightarrow 8\mathrm{M(NO_3)_2} + 6\mathrm{H_2O} + 2\mathrm{NH_3}$$

(5) When hydroxylamine is one of the products. NH_2OH can be expressed algebraically thus:

$$2\mathrm{NH_2OH} = \mathrm{N_2H_2} + 2\mathrm{H_2O}$$

but

$$\mathrm{N_2O}_x = \mathrm{N_2H_{-2x}} + x\mathrm{H_2O}$$

In order to obtain an expression for N_2H_2, -1 is substituted for x:

$$\mathrm{N_2H_2} = \mathrm{N_2O^{-}_1} + \mathrm{H_2O}$$

therefore,

$$2\mathrm{NH_2OH} = \mathrm{N_2O_{-1}} + 3\mathrm{H_2O}$$

The equation for a divalent metal is:

$$6\mathrm{M} + 14\mathrm{HNO_3} \rightarrow 6\mathrm{M(NO_3)_2} + (7\mathrm{H_2O} + \mathrm{N_2O_{-1}}) \rightarrow 6\mathrm{M(NO_3)_2} + 4\mathrm{H_2O} + 2\mathrm{NH_2OH}$$

[1] Atomic Energy Commission, Chemical Division, 53 Peddar Road, Bombay 26.

[2] MELLOR, J. W., "A Comprehensive Treatise on Inorganic and Theoretical Chemistry," Longmans, Green and Co., London and New York, **1947**, Vol. VIII, pp. 591–2.

THE CHEMICAL STUDY OF AN ANTIQUE ART OBJECT

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(Translated by Ralph E. Oesper)

THE judging of art objects from past epochs by antiquarians alone on the basis of stylistic studies and experience is no longer adequate in many cases to yield a verdict that is final in all respects. Purely scientific methods must be included before technically well-founded conclusions can be reached through examination of the specimen.[1] Since the majority of such examinations require the solving of chemical-analytical problems, in which the chief requirement is the minimum possible damage to the object under investigation, only microanalytical, microscopic, roentgenographic, and spectrographic methods are applicable as a rule.[2] As has been shown in many instances, the use of microanalysis has proved of exceedingly high value, not only in judging art objects but also in their preservation and restoration. In these latter cases, however, one or more chemical substances are applied to the art object and thus brought into contact with chemical substances that are already present and with which they most certainly must react. Consequently, if the conservator or restorer lacks the necessary knowledge of materials, damage may result which, if not visible immediately, may none the less appear in the course of years.

Purely technical examination may be necessary to supplement the stylistic study and the chemical-analytical investigation of art objects.

This paper reports the findings obtained with a helmet mask belonging to the collection of antiquities of the Kunsthistorischen Museum in Vienna.[3] This piece has been in the possession of the museum for about 80 years; it was found in Rumania in 1863. Nothing is known of its history and doubts of its genuiness have been expressed. Recently, several similar pieces were disinterred near Munich and the museum authorities have naturally wished to establish any time relationship between this find and the mask in their collection. The authors of this paper were asked to make a scientific study of the Vienna mask and, if possible, to establish its authenticity. The task proved to be laborious and required about half a year. The study was directed toward answering especially four questions: (1) What is the composition of the metal? (2) Is the mask an electrotype? (3) Is the present color the natural or an artificial patina? (4) What are the inside crusts—sand?

GENERAL DESCRIPTION

The very well-preserved mask (Figure 1) is about 23 cm. high and about 20.5 cm. wide at its maximum

Figure 1. Helmet Mask from the Antique Collection of the Kunsthistorischen Museum in Vienna

points. The exterior has a reddish-brown patina, and again and again shows (Figure 2) scarlike depressions but no hammer marks. The inner side has black-brown to dark olive green patina coating and shows distinct hammer marks (Figure 3) as well as pitting, but not to the same extent as the outer side. The oak leaf wreath, which crowns the part in the hair, is already broken through in several places; the eye openings are

[1] STREBINGER, R., "Interessante Beispiele der angewandten Mikrochemie," *Mikrochemie*, **36/37**, 805 (1951).

[2] STREBINGER, R., "Die Schaden an den Sarkophagen der Kapuzinergruft," *Oesterr. Z. Denkmalpflege*, **5**, 67 (1906).

[3] BENNDORF, O., "Antike Gesichtshelme und Sepulcralmasken, gefunden 1863 im Oltfluss bein Kloster Comani in Rumänien," *Denkschriften Akad. Wiss. Wien*, **28**, 223 (1878).

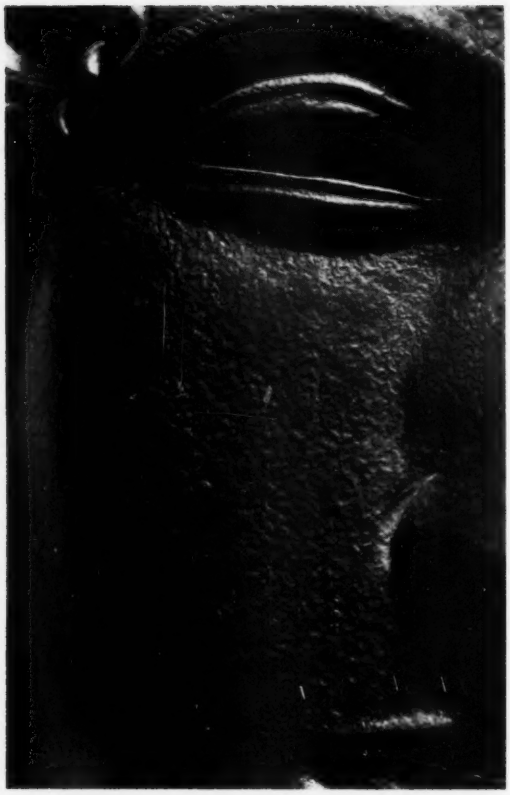


Figure 2. Part of the Cheek, Showing Scar-like Corrosion Pits

relatively sharp-edged. The entire mask has a strongly thickened edge. At a few places on the inside there is a sandlike coating. On both sides, in the ear regions, there are openings, obviously intended for binding cords.

The thicknesses of various parts of the mask are:

Oak leaf	0.65–0.8 mm.
Hair	1.00–1.3
Eyelids	1.2
Cheek (jaw bone)	1.5–20
Cheek	1.0
Chin	0.5–0.9
Edge, upper (hair part)	2.2–2.8
Edge, lower (neck)	1.7–2.5

CHEMICAL ANALYSES

Mask Metal. Samples, totaling about 20 mg., were taken from the region of the openings just mentioned. Qualitative microanalysis showed the presence of copper, zinc, tin, iron, lead, arsenic; phosphorus and antimony were detected spectrographically.

The material of the metallographic investigation was used in the quantitative analysis. The composition of the metal was found to be:

	Macroanalysis, %	*Microanalysis, %*
Copper	96	96.4
Zinc	1.8	1.7
Tin	0.9	1.0
Iron	0.2	0.2

The remainder is distributed among the above impurities, including oxygen.

Sandlike Crust. The extremely adherent coating is pure SiO_2. The microscopic picture showed round, abraded grains.

METALLOGRAPHIC STUDIES

All of the metallographic studies described below were made on polished cross sections of the mask foil or the comparison specimens, either unetched or etched with cupric-ammonium chloride solution.

Mask. The sample (about 300 mg.) exhibits numerous, irregularly distributed oxidic inclusions (Figure 4*a*). Some of these are circular and have a pale light-blue color, which becomes weakly reddish in the dark-field (sidelighting). The remaining inclusions, which are in the great majority, appear dark when observed in direct light and have an irregular angular shape. The arrangement of the inclusions indicates that rolled foil could hardly have been used, since, as will be shown later, rolled foil exhibits elongated inclusions, which are arranged chiefly in the direction of the rolling. The difference in the nature of the inclusions is particularly noticeable in Figure 5; the darker and lighter inclusions are then quite apparent. Figure 5*a* is a picture of the section in rectilinear incident light,

Figure 3. Inner Side of the Mask with Distinct Hammer Traces and Corrosion Scars

while Figure 5*b* shows the same area in the dark-field, where the darker inclusions light up brightly and the light-blue ones only become more visible in sharply distinguished contours (particularly along the line *a* . . . *b*).

The etched section (Figure 4*b*) shows, in addition to the oxidic inclusions, the structure of lightly alloyed copper (α-structure).

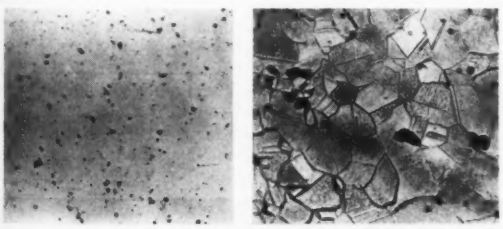
Several old pieces of sheet copper were studied for comparison:

(1) *Maria Theresia penny* (1717–80): The section has numerous light and darker inclusions (Figure 6*a*), whose arrangement in a definite direction is determined by the rolling process. In this specimen, the roundish pale-blue Cu_2O inclusions predominate; they glow reddish in the dark-field. The etched section (Figure 6*b*) shows the characteristic α-structure.

(2) *Copper Dish* (*Seventeenth Century*): This comparison object (Figure 7) yielded the same observations as (1).

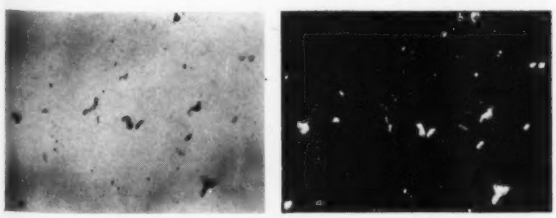
(3) *Copper Vase* (*1764*): Whereas the pale-blue inclusions predominated in (1) and (2), the darker ones are in the majority in this specimen (Figure 8). Furthermore, no uniform direction of their arrangement could be found; a fact doubtless due to the necessary hammer work that followed the forming of the vase.

The metallographic studies of the material, of the mask as well as the three comparison objects from the seventeenth and eighteenth centuries, reveal the pale-blue inclusions, which light up red in the dark-field, a quite characteristic feature of cuprous oxide inclusions and also of other oxides that appear dark under the microscope. The copper crystals that appeared after etching are somewhat smaller in the case of the mask,



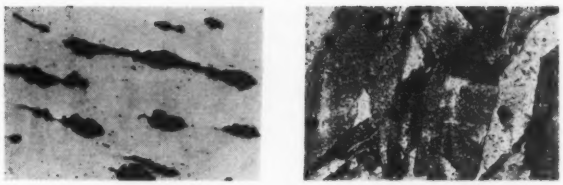
(*a*) (*b*)

Figure 4. Mask Metal, Polished
(*a*) Unetched (50×) (*b*) Etched (320×)



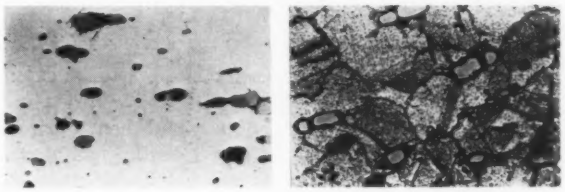
(*a*) (*b*)

Figure 5. Mask Metal, Polished, Unetched (188×)
(*b*) Same region as (*a*) taken with dark-field illumination



(*a*) (*b*)

Figure 6. Maria Theresia Penny, Polished (285×)
(*a*) Unetched (*b*) Etched

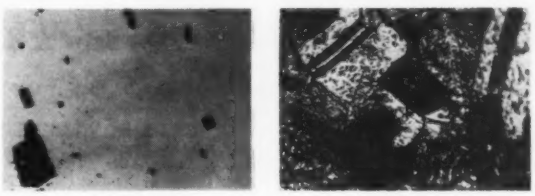


(*a*) (*b*)

Figure 7. Old Copper Vessel (Seventeenth Century), Polished (285×)
(*a*) Unetched (*b*) Etched

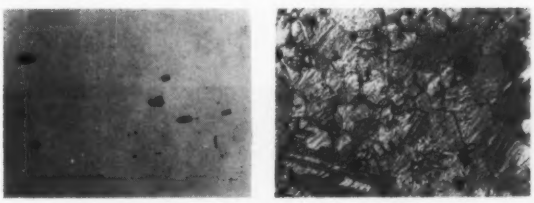
which indicates that there was no protracted heat treatment when it was formed by hammering.

In addition, a modern copper foil was similarly examined for comparison. This foil, prepared from electrolytic copper by melting and rolling, has only isolated



(*a*) (*b*)

Figure 8. Copper Vase (1764), Polished (285×)
(*a*) Unetched (*b*) Etched



(*a*) (*b*)

Figure 9. Copper Foil (Modern), Polished (285×)
(*a*) Unetched (*b*) Etched

larger cuprous oxide inclusions, of the characteristic form and color; the copper crystals (α-structure) are smaller than in the mask (Figure 9).

(4) *Cathode of a Copper Coulometer:* A section was prepared from pure electrolytic copper for additional comparison purposes (Figure 10). The difference between the galvanic coating and the cathode core is very distinct. The latter was prepared by melting and

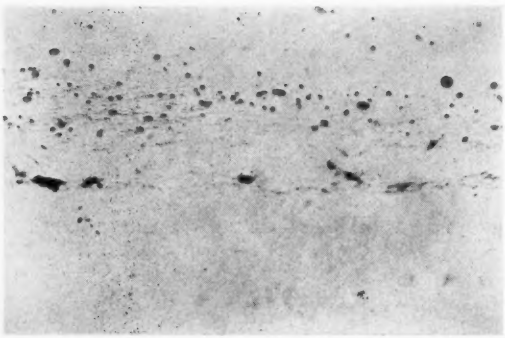
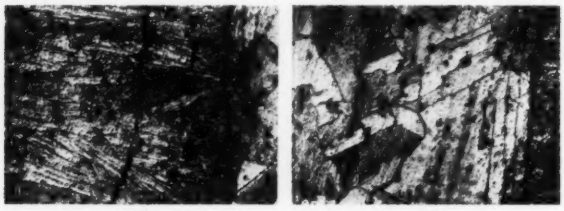


Figure 10. Copper Coulometer Cathode, Polished, Unetched (513×)

casting purest electrolytic copper and hence is, of course, interspersed with cuprous oxide inclusions. The latter are entirely absent in the galvanic coating. The difference is still more evident between cast electrolytic copper and electrolytically precipitated copper, if the etched sections are examined. Figure 11 shows



(a) (b)

Figure 11. Copper Coulometer Cathode, Polished, Etched (215×)
(a) Region of the electrolytically deposited copper
(b) Region of the copper nucleus

clearly the radiating structure of the deposited electrolytic copper and the characteristic α-structure of the cast and rolled electrolytic copper foil as opposed to the core. In addition to the dividing line between the zones, the current-interruption lines can also be seen. In this connection, note should be taken of the fact that when the current was again sent through the system, the copper continued to build up its electrolytic accumulation at the same place in the same direction.

STUDY OF THE FINE STRUCTURE

Since rolled copper foil has been in use since the sixteenth century,[4] a study of the fine structure of the material of the mask was made by means of X-rays of uniform wave length (chromium radiation). If the mask had been constructed from rolled foil, this would be made manifest by an irregularity in the blackening of the two lines of the glide plane of the copper lattice, a so-called rolling texture. However, the reflected ray picture shows nothing of the kind; *i. e.*, the blackening of the lines was perfectly uniform (Figure 12). In order to substantiate this finding, two-thirds of the total thickness of a second sample of the mask material was etched away with nitric acid and a reflected ray picture taken of the remaining portion. Once again, no texture could be seen, but there was a resolution of the lines into individual points (Figure 13), which can be explained as due to a recrystallization of the material.[5]

CONCLUSIONS

The four questions posed at the start can now be answered on the basis of the experimental findings:

(1) *Composition of the metal:* The material used for the construction of the mask was a relatively pure copper, containing various metal admixtures, which were certainly not added intentionally but were impurities introduced through the process of obtaining the metal from the ores. The use of electrolytic copper can be rejected without hesitation; only copper obtained by metallurgical methods need be considered.

(2) *Is the mask an electrotype?* The analytical findings themselves exclude the use of electrolytically deposited copper and the metallographic studies give proof that the mask is neither a galvano nor a partial electro deposit.

[4] A rolling mill for copper, lead, and other soft metals was described by Leonardo de Vinci in "Codex Atlanticus."

[5] Acknowledgment is made here for the aid of Frau Dr. Castelliz in carrying out the study of the fine structure.

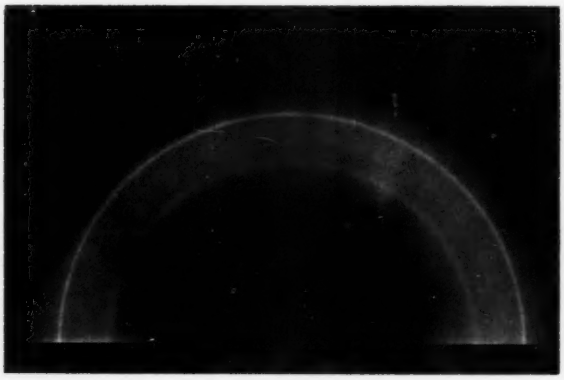


Figure 12. Reflection X-ray (Cr Radiation) Photograph of Surface of Mask Metal

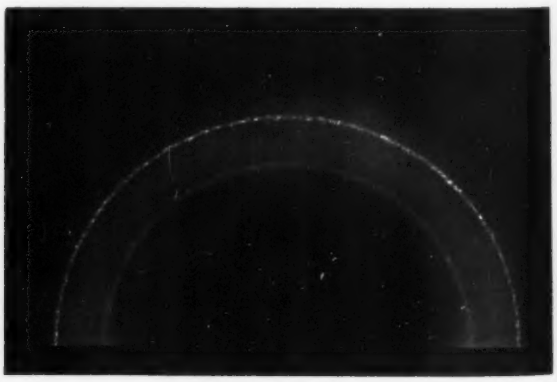


Figure 13. Reflection X-ray Photograph of Mask Foil, Etched off to a Third Original Thickness

(3) *Is the present color a natural patina or has it been produced artificially?* Since neither the inner nor the outer surface gave any chemical or microscopic evidence pointing to an artificial patination, the present color of the mask, both on the inside and outside, must be accepted as a naturally acquired patina. In the writers' opinion, the corroded layers were removed with utmost care about 60 years ago from both the inner and outer surfaces, and the traces of these corrosions may now be seen as the pocklike pits on both surfaces. This conclusion is strengthened by the results of the fine structure analysis. Consequently, the brown-red patina of the outside can be taken to be about 60 years old.[6] The brown-black patina on the inner side, for the most part, doubtless dates from the time of the manufacture of the mask, since the black copper oxide formed at that time offers high resistance to other corroding influences.

(4) *What are the interior crusts?* The slight crustlike coating is sand. Its tenacious adherence leads to the conclusion that it has acted on the mask wall for a long time, possibly under pressure, and has thus entered into combination with the previously corroded base. The rounded grains indicate river or sea sand.

Attempt to Reconstruct the Method of Producing the Mask. The work piece, consisting of metallurgically obtained copper, was brought to approximately the size of the surface of the mask by hammering the metal while it was maintained at a moderately high temperature. The overhanging material was cut off during this operation, so that finally there resulted a basic hollow form in the shape of the mask. The edge was thickened into a raised border by hammering; the hammer marks can still be seen on the inner side of the mask. The hammering was done during a heat treatment that was not unduly prolonged. After the surface was finished, it assuredly was quite smooth. The scarlike pits, on both the inside and the outside, are the results of a long-continued corrosion in which the copper was converted into basic salts (verdigris). This corrosion was not uniform over the entire mask; it was particularly visible in the hair portions of the mask (Figure 14) and on both the entire inside and outside surfaces (Figures 2 and 3). At some later date, the mask was carefully cleansed of the basic copper compounds, dirt, etc., and since then has acquired its present lovely brown-red copper patina through the action of natural agencies.

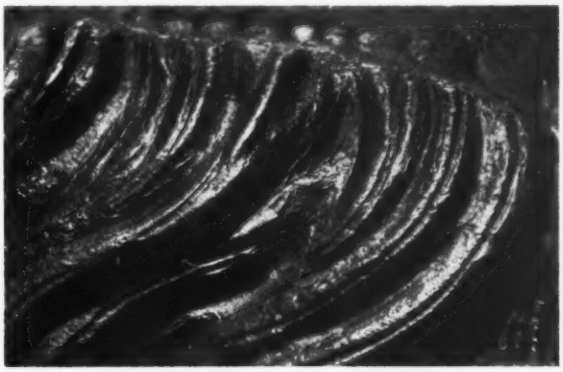


Figure 14. Part in Hair of Mask Showing Distinct Corrosion Pitting

If stylistic critical studies and the results of comparisons carried out by expert art historians should point to the mask being an antique, the scientific findings will fit this conclusion with no difficulty whatsoever.

[6] See Benndorf's paper (footnote 3) where it is stated (p. 322) "It has been reported to me that it could be seen brightly polished at the Paris World Fair of 1867." According to a communication from Professor Eichler, Director of the Kunsthistorischen Museum in Vienna, it is entirely possible that the mask was free of a surface layer at the time of its discovery, as is frequently the case with antique articles taken from rivers.

ACKNOWLEDGMENT

The authors wish to express their appreciation to Dr. Josef Nagler, Direktor des Technischen Museums in Vienna, Dozent Dr. E. Holzmair, Leiter der Bundessammlung von Medaillen, Münzen, und Geldzeichen in Vienna, and Professor Dr. Leopold Schmidt, Leiter des Museums für Volkskunde in Vienna, for placing at their disposal the old copper objects and coins required for the comparison studies.

ERRATUM

On page 640 of the December, 1952, issue, the price of B. Pullman and A. Pullman's "Les théories électroniques de la chimie organique" is given as 2800 Frs. It should be 5800 Frs.

SMALL-SCALE TECHNIQUES IN THE TEACHING OF ORGANIC CHEMISTRY[1]

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MICRO- and semimicro chemical techniques, mandatory where the availability of materials is small, have become increasingly popular for general usage. Saving in materials, bench room, storage space, power, and time, together with a reduced laboratory fume-level and a lessened accident-risk, are features which render small-scale working attractive for teaching. Much progress has been made toward the general adoption of semimicro methods in qualitative inorganic analysis; that organic chemistry is not being neglected is shown by the existence of several textbooks describing small-scale operations in this field of work (*1*, *2*, *8*, *10*).

Even in teaching, however, it is neither likely nor desirable that small-scale working will displace conventional organic procedures, but rather act as a valuable and convenient supplement. A student trained solely in gram- and decigram-scale working would prob-

[1] Presented at a meeting of the London Section of the Science Masters' Association, January, 1953, and at a meeting of the Birmingham University Chemical Society, February, 1953.

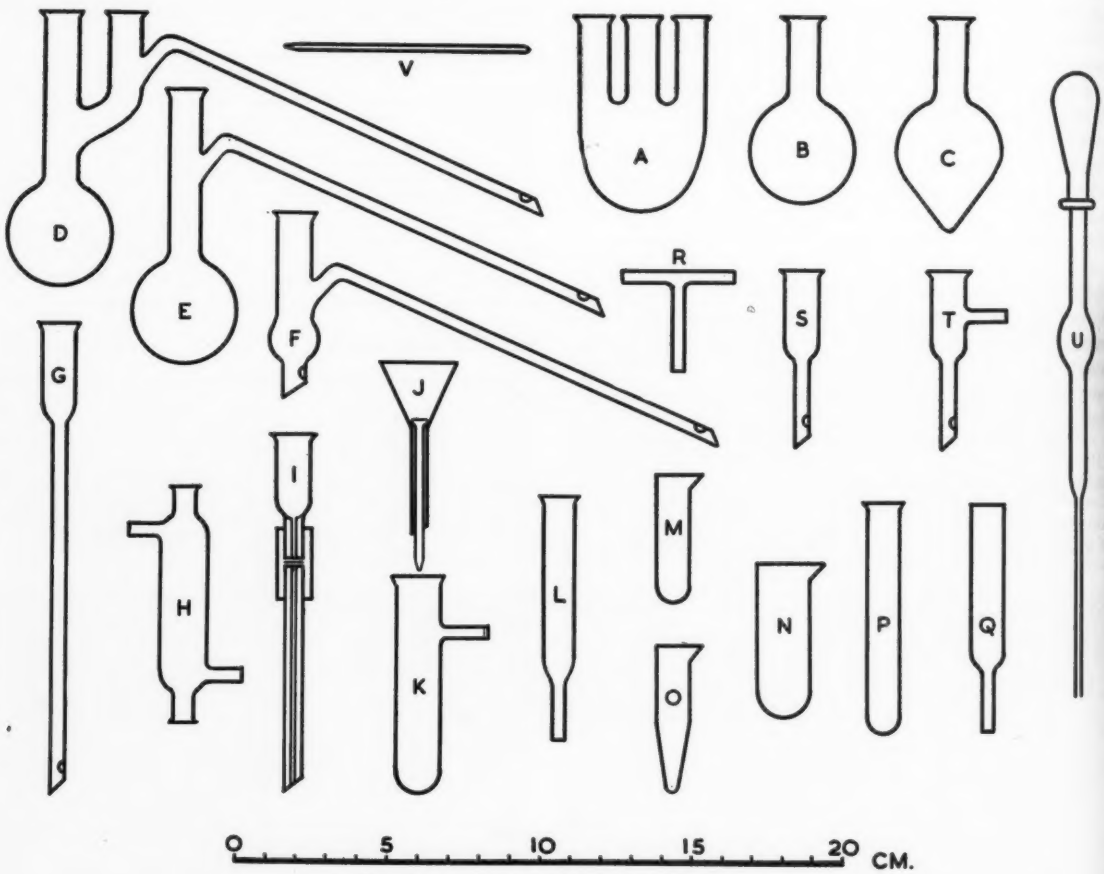


Figure 1. Student's Standard Set of Apparatus

A, Reaction flask
B, *C*, Reaction and distillation flasks
D, *E*, Distillation flasks
F, Still-head for reaction flasks
G, Condenser inner
H, Condenser jacket
I, Schwinger filter
J, Willstatter "nail" filter
K, Filtering tube
L, Drying tube
M, *N*, Crystallizing tubes
O, Receiver
P, Test tube (6 off)
Q, Adaptor
R, Tee-piece
S, *T*, Filtering adaptors
U, Teat pipet
V, Glass rod

ably not do very well if asked to prepare the large batch of starting material often needed for a multistage organic synthesis. Apart from any difficulties arising from unfamiliarity with the larger apparatus, the "scaling-up" of a preparation, involving its own problems such as heat transfer, agitation, etc., is often quite as difficult as "scaling down." Although small-scale operations are introduced very early into our courses in organic chemistry, they are therefore used only when convenient and never slavishly.

Standard sets of small-scale apparatus (Figure 1) are issued to students in separate numbered boxes of dimensions $12^1/_2 \times 8^1/_2 \times 2^1/_{16}$ in. Besides being partitioned to allow the contents to be easily checked, these lidless boxes are arranged to stack, so that a dozen or more may be stored in the corner of a cupboard.[2] Made from 2- × $^3/_8$-in. planed hardwood strip cut to length in a few simple jigs and jointed by gluing and tacking, these boxes, which have bottoms of $^1/_{16}$-in. black plastic sheet, may be rapidly put together; one of our technicians produced a batch of 20 in less than two days. Additional apparatus, either spare or of type less frequently needed, is stored in similar boxes and issued on request. Heavy borosilicate glass is used throughout, neck and stem diameters being standardized as far as possible to permit the interchangeable usage of a single size of bored cork. Used in conjunction with ordinary stands and bossheads, the simple spring clamps (*14*) shown in Figure 2 not only permit rapid assembly and dismantling but also minimize breakage. Treated as consumable items, these clamps have hardwood stems about 6 in. long which are slotted as shown at (*a*) to accommodate spring "Terry clips" of suitable sizes.

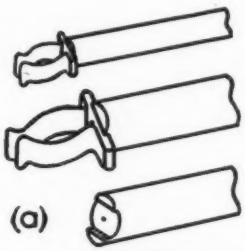
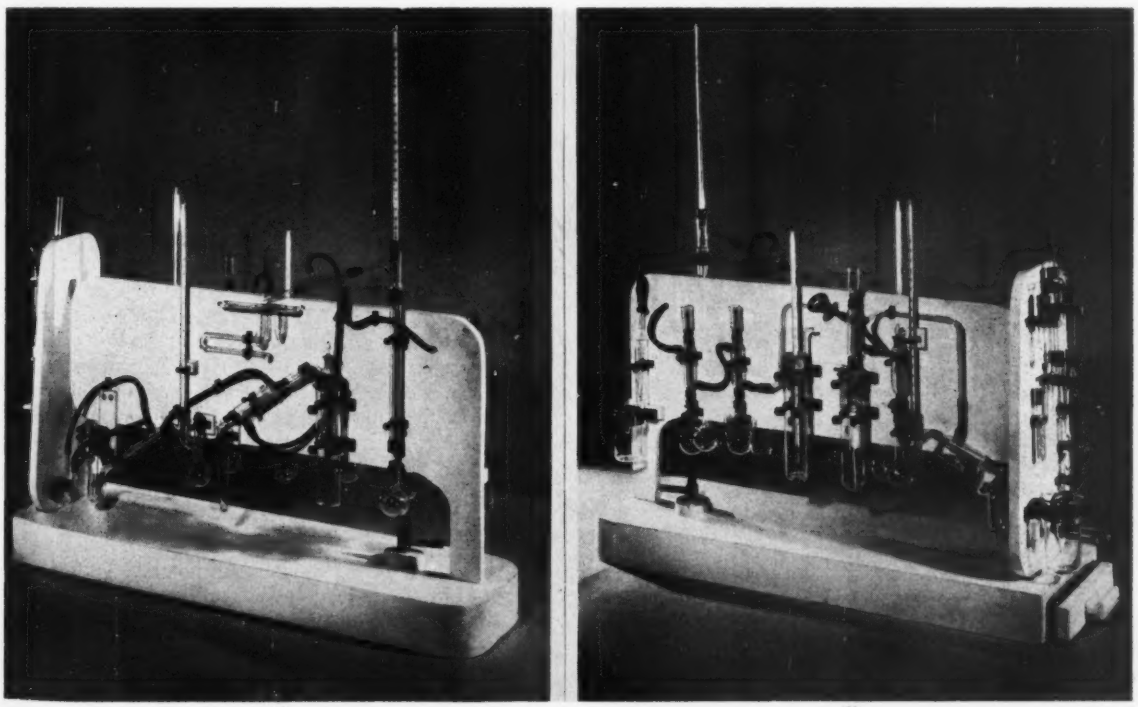


Figure 2. Simple Spring Clamps

For demonstration purposes, it is convenient to have compact permanent assemblies for the common operations of organic chemistry, such as fractional and vacuum distillation, continuous extraction, etc. Figure 3 shows a double-sided unit designed for this purpose (*16*); when out of use, the thermometers are stored along with additional apparatus in the long drawer in the base. Apart from its handiness as a teaching aid, this unit may prove useful in mobile laboratories. A compact set of standard cone-jointed apparatus, clip-mounted in a fold-up case for easy transport (*16*), is convenient for the demonstrator's private use. A further example of current British practice is the set of organic apparatus for gram-scale working recently described by Claret (*3*).

The simple but highly effective "Willstatter nail" filter (shown at *J* in Figure 1) is very popular with our

[2] These, and many pieces of apparatus described in this paper, were demonstrated at the International Congress on Analytical Chemistry, Oxford, England, September, 1952.



(a) (b)

Figure 3. Demonstration Setup. (a) First Side; (b) Second Side and End

students and is used under light suction. Filter-paper discs are cut with a corkborer of appropriate size. Several "nails" of differing head size may be made for a single funnel, so that a paper of diameter appropriate to the amount of solid to be collected may be employed.

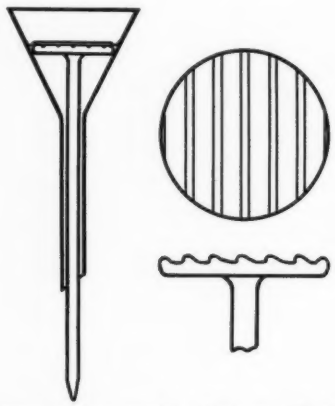


Figure 4. Corrugated-head Filtering "Nail"

If the head of the "nail" is corrugated as shown in Figure 4, drainage of filtrate over the entire area of the paper can occur. Although this modification is quite unnecessary in the small sizes normally used, it does

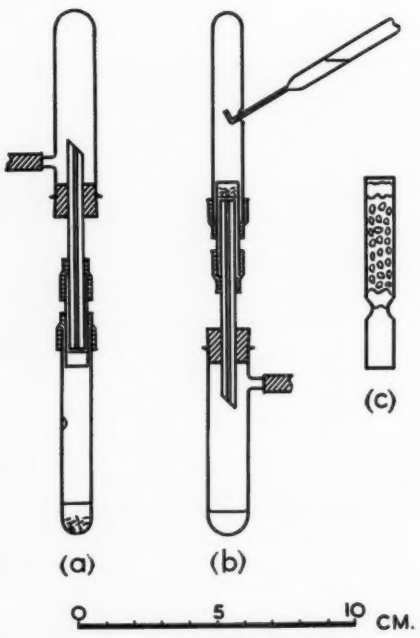


Figure 5. Use of the King Filter

permit the making of a filter large enough to accommodate a 25-mm. paper, thus providing a cheap substitute for a Witt plate or a Hirsch funnel. The corrugations are easily produced by pressing the hot glass on the surface of an old single-cut file or rasp of the coarsest grade. Where the amount of solid is small, the Schwinger filter (*I*, Figure 1) is particularly useful. The solid collects as a pellet above the tiny filter-paper disc in the throat of the filter; after dismantling, the pellet may be expelled bodily by a snugly fitting rod.

The preparation of the acetyl derivative of a primary amine is used to illustrate other separation techniques (*14*).

Transfer one drop of aniline to a micro test tube and add 1 ml. of water and one drop of acetic anhydride. Mix well by shaking, then add 8 to 10 drops of 4 *N* sodium hydroxide solution, when the crude derivative should separate. Centrifuge gently, discard the liquid, stir the solid with a few drops of water, centrifuge, and discard the washings. Add a few mg. of fine charcoal or kieselguhr and 0.5 to 1 ml. of water, heat gently until the derivative dissolves, then centrifuge immediately. Using a warmed teat pipet, at once transfer the hot solution to a micro test tube having a 4-mm. hole midway in the wall. Allow to cool, when crystallization should occur.

Place a filter-paper disc in the cup of a King filter (*6*), apply gentle suction to the lower end and, by means of a sleeve of rubber tubing, attach the filter to the mouth of the test tube as shown at (*a*) in Figure 5. Agitate the test tube and, keeping the hole on the upper side, carefully invert and rotate slowly so that the contents flow into the cup, leaving the crystals evenly deposited on the filter disc. Complete the transference with about 0.5 ml. of water delivered from a right-angled pipet as shown at (*b*); a series of puffs of air directed upon the rim of the filter-cup will dislodge any particles trapped there. Replace the test tube with a calcium chloride drying tube as shown at (*c*), maintain moderate suction for 5 minutes, then eject the dried crystals onto a watch glass and determine the melting point.

Since stirring, where necessary, of organic reaction mixtures often has to be maintained for considerable periods, agitation by bubbling air, nitrogen, etc., through the liquid (a useful expedient in micro-volumetric work) is of little avail, owing to loss of volatile constituents. Where the viscosity of the reaction mixture is not high, various forms of magnetic stirrers (*12*) are useful. The body of the rotary type shown in Figure 6 is a hardwood block, in the upper surface of which is bored a smooth-walled hole about 55 mm. in diameter and 25 mm. deep. The airtight cover plate *B* is of $^1/_{16}$-in. white plastic sheet, the spindle *C* being a portion of a sewing needle which is driven squarely with point upward into the center of the bottom of the hole. Cut from a 50-mm. diameter cork, the rotor *D* is provided with rachet-like teeth as shown at (*a*). An alternative but not quite so effective rotor shown at (*b*) and (*c*) is a plastic reagent-bottle cap, around the circumference of which is cemented a strip cut from a corrugated rubber mat. The $^3/_4$-in. diameter Alnico magnet[3] *E* is secured to the rotor by the brass bolt *F* in which a blind hole is drilled to form a bearing for the needle point. The jet *G* is merely a $^1/_{16}$-in. hole drilled in the side of the block as shown. To facilitate entry of air, the hole is opened out to about $^1/_4$ in. for two-thirds of its length. Application of filter-pump suction to the connection *H* causes the rotor to spin rapidly.

[3] James Neill (Sheffield), Ltd., Sheffield, England. Type M4776.

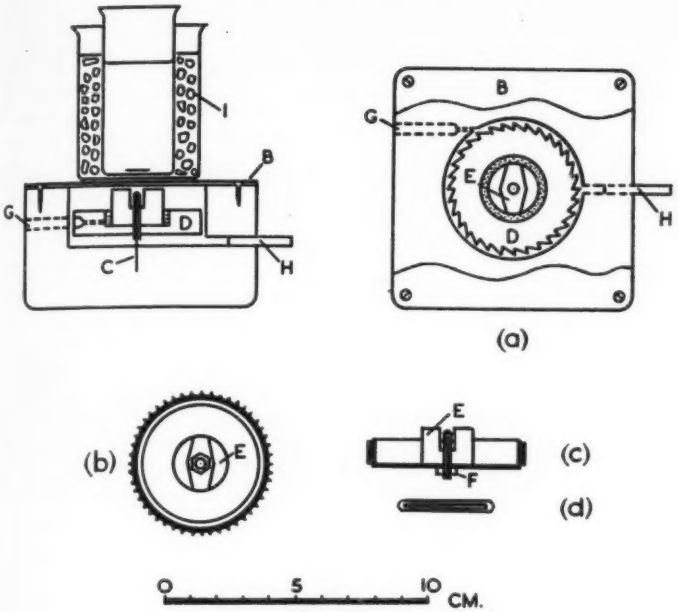


Figure 6. Miniature Magnetic Bar Stirrer

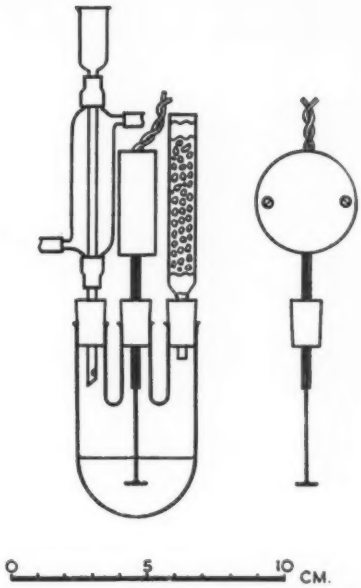


Figure 7. Miniature Reciprocating Stirrer

The stirrer bar, about 10 mm. long, is made by sealing a few strands of fine iron wire or a portion of a sewing needle within melting-point tubing as shown at (*d*). To avoid bulging or actual blowing out during the closure of the second end, it is advisable to thoroughly heat the *whole* of the partly finished stirrer bar by holding in an old pair of tweezers just above the blowpipe flame, taking care not to soften the glass. The open end is then touched with the flame to seal it. It may take 10 minutes to make the first satisfactory bar; after this, a dozen can be made in half an hour. Since the bar may be easily driven through a double beaker thickness, the ice-bath arrangement *I*, so useful for diazotizations, etc., may be employed as required.

Where the curvature of the reaction vessel is too great for the proper operation of the bar-type stirrer, the miniature solenoid-operated reciprocating stirrer shown in Figure 7 is useful (*15*). The body of the unit consists of two reagent-bottle caps bolted mouth to mouth; some models carry a small knob for adjusting the stroke while running. Being light and very compact, this stirrer may be supported directly in the neck of a micro reaction flask as shown.

Whereas sealed-tube macro-scale experiments are not generally favored, micro- and semimicro pressure reactions are often convenient and we have found that, with proper precautions, they are safe even under examination conditions. The employment of small sealed tubes, dating back at least as far as 1883 (*5*), is certainly no novelty. Although an ordinary laboratory oven may be used for heating, we prefer to use simple thermostatically controlled heating blocks of the type shown in Figure 8. The "block" is a tin can containing small nails (*13*); pockets for the thermometer and for the reaction-tube holders are old corkborers which are tightly plugged at the lower ends with fine steel wool. The tubeholders are similarly plugged corkborers which slide easily into the pockets and have wire lifting loops. As the working temperature is approached, expansion of air in bulb *J* cuts off that portion of the gas supply normally passing through jet *K*. The screw clamp *L* allows the temperature to be adjusted between the approximate limits of 100° to 250°C. An improved

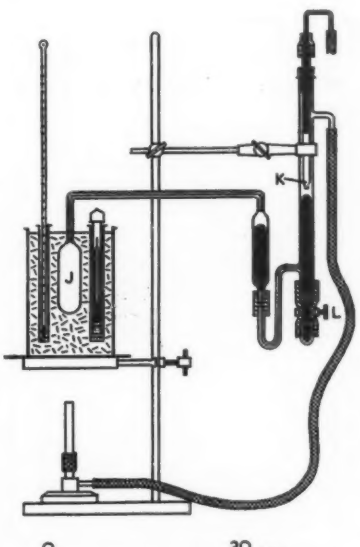


Figure 8. Thermostatically Controlled Heating Block

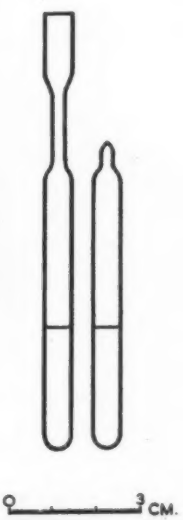


Figure 9. Tube for Pressure Reactions

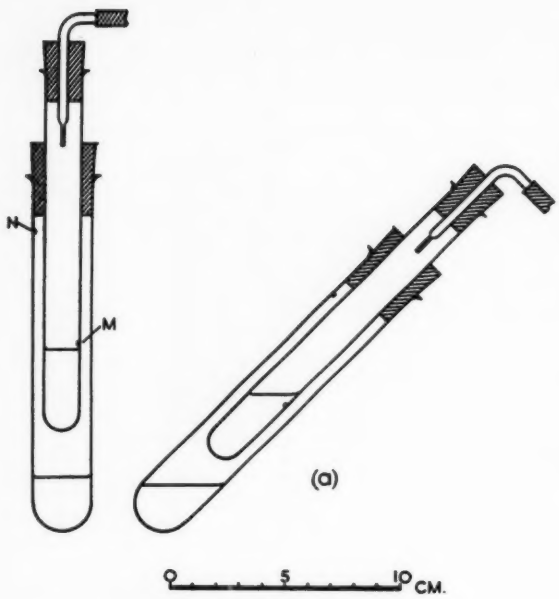


Figure 10. Air-flow Apparatus

version of this device, incorporating a compact regulator and a magnetic gas valve, is being developed (*16*).

The dinitration of chlorobenzene and subsequent pressure ammonolysis to 2, 4-dinitroaniline (*4*) is a simple two-stage preparation illustrating sealed-tube technique. Having performed the first stage in a test tube on more or less conventional lines, the ammonolysis is performed as follows (*7*):

From a 100-mm. length of 4-mm. tubing (wall thickness 1 to 1.5 mm.) construct a reaction tube as shown in Figure 9. Dissolve 30 mg. of 2,4-dinitrochlorobenzene in 10 drops of warm methylated spirit, transfer about half to the cup of the reaction tube and, by shaking or centrifuging, cause the liquid to enter the tube. Similarly introduce the other half of the liquid, completing the transfer with 2 or 3 drops of spirit. Follow with 10 drops of concentrated ammonia (sp. gr. 0.88) and make a strong seal. Heat at 150 to 160°C. for 30 minutes and allow to cool vertically. Cut off the upper end of the tube and discharge its contents into a crystallizing tube, completing the transfer with a jet of water after cutting off the other end. Stir well, filter through a Schwinger filter, and expel the solid into a 30- × 12-mm. crystallizing tube, completing the transfer with 5 to 10 drops of methanol. Heat very carefully until complete solution results, then add water drop by drop until just permanently turbid. Clarify with a drop of methanol and allow to cool, finishing off with 5 minutes in ice. After using the mother liquor once to complete the transference of the crystals to a Schwinger filter, wash with 1 drop of 20 per cent (v/v) methanol and suck dry after attaching a drying tube. The average yield under class conditions is in the region of 50 per cent, but this figure can be pushed up to over 70 per cent by a practiced worker.

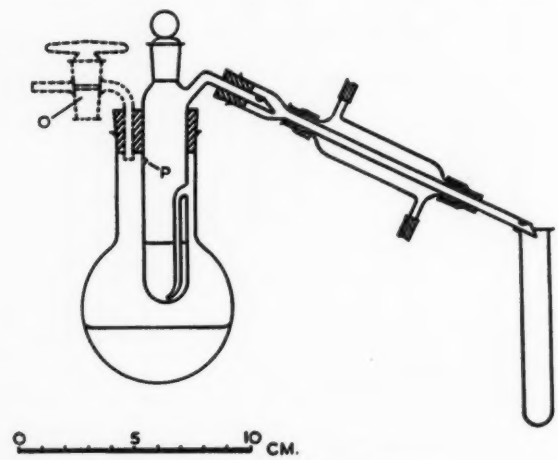


Figure 11. Steam-distillation Apparatus

The hydrolysis of 0.5 ml. of ethyl benzoate and identification of the products is used to illustrate saponification (*14*). Benzoic acid is, of course, easily isolated from the reaction mixture; the ethanol, obtained as a dilute distillate, is identified by acid dichromate oxidation to acetaldehyde, which is aspirated into 2, 4-dinitrophenylhydrazine solution. A simple and compact

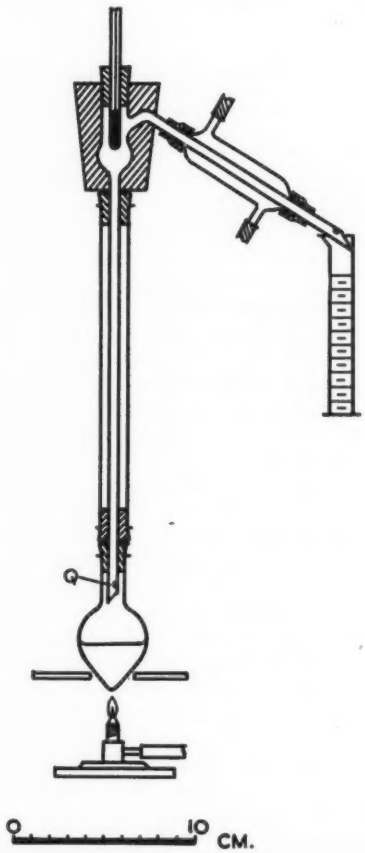


Figure 12. Simple Fractional-distillation Apparatus

air-flow apparatus for the oxidation and precipitation stages is shown in Figure 10. The distillate and oxidizing agent is placed in the outer vessel and the inner tube, which has a 1-mm. hole *M* blown in the wall, is

filled to the level shown with the precipitant solution. A slow stream of air, entering through the groove *N* in the larger cork, is drawn through the apparatus, which is then tilted as shown at (*a*) to cause 1 to 2 bubbles per second to form in the precipitant solution. The outer vessel is then gently heated for about 5 minutes, during which the yellow derivative should have appeared in the inner tube. Before discontinuing suction, the apparatus is restored to the vertical position.

The mononitration of phenol and the isolation of the *ortho* isomer is used to illustrate both the handling of a tarry reaction mixture and the use of the Pozzi-Escot steam-distillation apparatus (Figure 11). Although sucking back can be prevented by the timely opening of stopcock *O*, a simpler and foolproof alternative, by which the stopcock is dispensed with, is the provision of a tiny hole *P* in the wall of the distillation vessel. By-passing of steam then naturally increases the volume of distillate, but this is not serious unless maximum yields are of more than usual importance.

Fractional distillation is introduced by use of the simple air-jacketed 4-mm unpacked column shown in Figure 12. A small hole *Q*, blown at the foot of the column, prevents the formation of a slug of liquid which might then be blown out of the column. The lagging in the region of the thermometer bulb is a split, suitably shaped cork. Although the maximum efficiency obtainable with a simple narrow tube (*9*) is hardly to be expected with this rather crude arrangement, the student cannot only obtain a reasonable distillation curve (Figure 13), but can also clearly see all stages of the operation of the column. The drastic reduction in efficiency resulting from excessive throughput is clearly brought out by the results obtained by any student who cannot "hurry slowly."

Although ultimate analysis is not attempted until later, various other quantitative experiments are included quite early in the course. Typical is the semimicro determination of the equivalent of an acid by titration and by ignition of the silver salt. For molecular weight determination, the Rast method is naturally used extensively; the vaporimetric technique is illustrated either by the use of a simple, very rapid Victor Meyer-type apparatus (*17*) or by a Dumas experiment. In the latter case, a 10-ml. ampoule which, being flat-bottomed, sits nicely on the balance pan, is used to construct the bulb. For ebulliometric molecular weight work a simple Cottrell-type apparatus is employed (*11*).

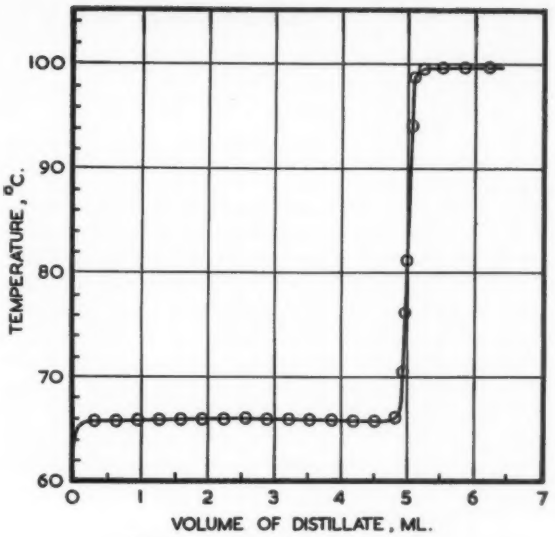


Figure 13. Typical Distillation Curve

Charge, 5 ml. methanol and 5 ml. water. Time from commencement to first runnings, 17 minutes. Rate of distillation, 0.11 ml. per minute.

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(2) Cheronis, N. D., and J. B. Entrikin, "Semimicro Qualitative Organic Analysis," Thomas Y. Crowell Co., New York, **1947**.
(3) Claret, P. A., *Chemistry & Industry*, **1952**, 1147.
(4) Clemm, C., *J. prakt. Chem.*, **1**, 170 (1870).
(5) Dreschel, E., *ibid.*, **27**, 422 (1883).
(6) King, E. J., *Biochem. J.*, **33**, 949 (1939).
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(10) Schneider, F., "Qualitative Organic Microanalysis," John Wiley & Sons, Inc., New York, **1946**.
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(12) Stock, J. T., and M. A. Fill, *Chem. Age*, **63**, 296 (1950).
(13) Stock, J. T., and M. A. Fill, *Analyst*, **76**, 497 (1951).
(14) Stock, J. T., and M. A. Fill, *School Science Rev.*, **33**, 331 (1952).
(15) Stock, J. T., and M. A. Fill, *Industrial Chemist*, **29**, 56 (1953).
(16) Stock, J. T., and M. A. Fill, unpublished experiments.
(17) Stock, J. T., and C. Heitler, *School Science Rev.* (in press).

A symposium on "Do's and Don'ts in Chemistry Building Construction" is being held at the Chicago meeting of the American Chemical Society. Anyone interested in presenting a paper should get in touch with Dr. A. H. Kunz, University of Oregon, who is organizing the symposium.

EDMUND DAVY

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Tulane University, New Orleans, Louisiana

MUCH has been written of the brilliant work of Humphry Davy, and in some accounts of his contributions to the development of chemistry in the early nineteenth century he has been given credit for investigations that were carried out by his less well-known cousin, Edmund Davy. Dr. John Davy, brother of Humphry, and Edmund Davy were extravagant in their praise of this unique genius so closely related to them. This admiration is not surprising for Humphry Davy had generously helped both of them to achieve success in their work. It might be of interest to note that neither Paris nor John Davy mention Edmund Davy in their lives of Sir Humphry Davy.[1]

Edmund Davy, son of William Davy, was born at Penzance, in Cornwall, in 1785, where he obtained his early education.[2] Humphry Davy was seven years older than Edmund, and had been professor of chemistry at the Royal Institution for almost three years when he used his influence to have Edmund appointed operator and assistant in the laboratory. The elder Davy demanded close attention to order in the laboratory from his assistants although he generally neglected this detail himself. Edmund Davy remained in London with the Royal Institution from 1804 until 1813. He was also the superintendent of the mineralogical collection of the Royal Institution.

The young man was, no doubt, present as the assistant at many of the very popular Friday evening lectures of Humphry Davy, four of which were attended by Michael Faraday in the spring of 1812. Faraday, it will be remembered, also through the influence of Humphry Davy, received an appointment at the Royal Institution as an assistant. These lectures, open to the public, made an impression on Edmund Davy which he carried with him after he left the Royal Institution, for he used his influence later to establish popular courses of lectures in most of the Irish provinces.

In 1813 Edmund Davy was unaminously elected professor at the Royal Cork Institution, possibly through the influence of Humphry Davy who had delivered a course of lectures in Dublin two years previously. In 1826, he became professor of chemistry of the Royal Dublin Society. He remained in Dublin until his retirement. He died on November 5, 1857, ten years before the death of Michael Faraday.

During his lifetime, Edmund Davy was overshadowed by Humphry Davy, the toast of scientific circles in London and on the continent, and by John Davy, world traveler in government service. Humphry Davy must have appeared much as a hero to Edmund and for most of his life he followed in the path of his cousin. Many of his investigations were centered around the ideas of Humphry Davy and when he thought that his experiments substantiated claims made by Humphry, he did not hesitate to say so. In 1812, while he was still at the Royal Institution, his first three papers were published, two of which were concerned with platinum, an element investigated concurrently by Humphry Davy. Both of the men published papers that year dealing with sulfur and phosphorus.

Edmund Davy is probably best known as the discoverer of acetylene although he obtained it in an impure state as a by-product.[3] In January of 1836 he was trying to prepare pure potassium by heating to a high temperature pulverized carbon with tartar (potassium hydrogen tartrate). From this he obtained a gray-brown mass which he treated with water and noted the evolution of a gas.[4] He showed the gas to be a compound of carbon and hydrogen and noted its burning with a brilliant flame, and its rapid reaction when brought in contact with chlorine. He proposed that the gas was formed out of a compound of potassium and carbon in the residue obtained by the heating process.[5] Although he correctly determined the carbon to hydrogen ratio of the gas, he did not establish its definite structure, nor did he give it its present name. This was left to Berthelot who, a quarter of a century later, showed that it can be obtained by the direct combination of carbon and hydrogen at the temperature of the electric arc, as well as in other ways. Calcium carbide, which is usually associated with the preparation of acetylene, was first prepared in 1840, four years after the work of Davy.

Much of Edmund Davy's early work was done with platinum and its compounds, and in this he was following a line of study closely related to that of Humphry Davy. In his discovery of the safety lamp in 1817, the elder Davy had come to the conclusion that in the presence of air a platinum wire through contact with the vapor of alcohol or ether, at temperatures below the

[1] PARIS, J. A., "Life of Sir Humphry Davy," Colburn, London, **1831**. DAVY, JOHN, "Memoirs of the Life of Sir Humphry Davy," Longman, Rees, Orme, Brown, Green & Longman, London, **1836**, 2 vols.

[2] "Dictionary of National Biography," Smith, Elder & Co., London, **1888**, Vol. 14, p. 185.

[3] WATTS, H. A., "A Dictionary of Chemistry," Longmans, Green & Co., London, **1883**, p. 1111.

[4] LIEBIG, J., *Ann.*, **23**, 144 (1837).

[5] The paper noting this discovery is listed in the Royal Society Catalogue of Scientific Papers, C. J. Clay & Sons, London, **1868**, p. 169, as being titled, "Notice of a new gaseous bicarburet of hydrogen, and of a peculiar compound of carbon and potassium or carburet of potassium." This paper was published in *Bril. Assoc. Rep.*, **1836**, Pt. 2, pp. 62–4.

ignition temperatures, can become glowing and that an acid is formed. He charged Faraday to determine the certainty of this. Faraday found this acid to be acetic acid. Daniell also analyzed this acid by the method of Gay-Lussac and Thenard and verified Faraday's findings.[6] At about the same time, Edmund Davy, in a published report, stated that a preparation of platinum made by him, which he called fulminating platinum, possessed the remarkable property, at ordinary temperatures, of making wine spirit (ethyl alcohol) glow so strongly that it ignited.[7] Oxidation of the alcohol to acetic acid took place so rapidly at the surface of the platinum that the latter was raised to incandescence.

While at the Cork Institution he reacted platinum sulfate with alcohol and produced a black powder which appeared to consist almost entirely of platinum. This powder reacted with alcohol vapor, and he found that "...the acid first noticed by Sir Humphry Davy (in his beautiful experiment of the ignited platinum wire, and since, more fully examined by Mr. Daniell) is produced." Davy then constructed a tinder-box, "...to procure immediate light by means of the powder." He also prepared an oxide of platinum and found it to be insoluble in aqua regia, "...a fact which seems to add additional support to Sir H. Davy's opinion respecting the action of aqua regia on platinum."[8] Sir Humphry Davy had been of the opinion that aqua regia did not form an oxide with platinum when the metal was dissolved, but merely caused its combination with chlorine. Edmund Davy stated that his findings supported Humphry Davy's claims, a matter in which they were correct. Platinum is dissolved by aqua regia, forming $PtCl_4$.

Döbereiner continued the investigation on the remarkable effects produced by platinum sponge and due to his interesting and spectacular demonstrations called the attention of chemists to the use of this form of platinum.[9] In 1821, Döbereiner showed that platinum black causes alcohol to combine with the oxygen of the air to form acetic acid and a product which he called "oxygen ether." Liebig later isolated acetal and acetaldehyde from this "oxygen ether." Döbereiner made an important discovery when he found that spongy platinum has the property of setting fire to a mixture of hydrogen and oxygen at low temperatures. Using this principle, he constructed a lighter which was popular until the advent of phosphorus matches.

In 1819, Edmund Davy published a paper on the solid excrement of the boa constrictor, in which he showed the existence of uric acid, although he was aware of the fact that Dr. Prout had previously discovered the presence of the acid in the boa excrement.[10] This paper, however, followed closely in time a similar work by John Davy and shows the attention which he gave to the work of his distinguished cousins.

He turned his attention to iodine and studied its action on various oils, including turpentine and lavender, linseed, olive, and castor oils. From this he concluded that, "...the action of iodine on volatile and fixed oils resembles that of chlorine on these bodies, a circumstance which serves to extend the analogies which Sir H. Davy has traced between iodine and chlorine in their chemical agencies."[11]

Davy published several articles on qualitative tests for the metals, including manganese and arsenic, and on the applications of electrochemistry. While at the Cork Institution, he suspected that the air of the Fever Hospitals might be impure and undertook experiments designed to discover if the composition of the air in the hospitals, devoid of proper ventilation, might be different from ordinary air. All of his experiments gave the composition of the air as the same as uncontaminated air, a fact which he definitely did not expect to find. He made some experiments on the buoys in Kingstown Harbour in order to try to find a method of protecting them from the action of sea water.

Always a favorite study with Davy was the application of chemistry to agriculture. In 1817, while studying the inferior crop of wheat produced the previous year, he arrived at the conclusion that "...the bad qualities of the new flour were connected with the production of saccharine matter in the grain."[12] He experimented with the hard water at Black Rocks, near Cork, and advised the residents as to how it could be made more applicable to domestic uses. He made a comparative study of the value of Irish and Virginia tobacco, and some experiments to detect and prevent frauds in the sale of skimmed milk. To aid the farmers of his district, he made many studies of the value of peat, charcoal, and lime as deodorizing agents. "An Essay on the Use of Peat or Turf as a Means of Promoting the Public Health and the Agriculture of the United Kingdom," was published for him by Hodges and Smith of Dublin in 1850.

Shortly after he became professor of chemistry of the Royal Dublin Society in 1826, he was elected a fellow of the Royal Society of London, a fellow of the Chemical Society of London, and an honorary member of the Société Française Statistique Universelle. During his life as a teacher and experimenter, he was always keenly interested in the spreading of chemical knowledge, and helped establish popular courses of lectures in the provinces, this idea being based on the enthusiasm with which the public lectures at the Royal Institution in London had been received. He personally gave more than 30 courses of lectures on chemical subjects, selecting especially subjects relating to applications of chemistry to agriculture.

Davy published 33 papers between 1812 and 1857, although the "Royal Society Catalogue of Scientific Papers" credits him with only 30 papers. When the

[6] GRAEBE, C., "Geschichte der organischen Chemie," Springer-Verlag, Berlin, **1920**, Vol. 1, p. 40.
[7] DAVY, E., *Phil. Mag.*, **56**, 332 (1820).
[8] *Ibid.*, p. 340.
[9] PRANDTL, W., "Johann Wolfgang Döbereiner, Goethe's Chemical Adviser," J. CHEM. EDUC., **27**, 176 (1950).
[10] DAVY, E., *Phil. Mag.*, **54**, 303–6 (1819).
[11] DAVY, E., *Phil. Mag.*, **59**, 208–11 (1822).
[12] DAVY, E., *Phil. Mag.*, **49**, 173 (1817).

government was changing the constitution of the Irish scientific societies, it recognized Davy's claims, by awarding him his entire salary upon retirement from his official position. The Royal Dublin Society requested him to continue the portion of his duties which related to agricultural chemistry after his retirement. After June of 1856 Edmund Davy suffered from ill-health until his death the following year at Kimmage Lodge, County Dublin.

ACKNOWLEDGMENT

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THE SHORT COURSE IN CHEMISTRY

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THE short course in chemistry is a course offered to special groups of students who need chemistry only incidentally to fulfill the requirements for a degree in another field. With the purpose of determining how widely such courses are offered, questionnaires were sent to 142 colleges having enrollments in excess of 3500 students. Replies from 118 colleges replying are tabulated. To find whether such courses are effective in preparing premedical students for medical school, questionnaires were sent to the 44 medical schools, 31 of which replied.

Thirty of the 118 colleges offer no short courses in chemistry. Eighty-eight colleges offer a total of 39 quantitative analysis, 64 organic, and 57 physical chemistry short courses.

The greatest number of short courses are offered in general by the state colleges, which also offer preprofessional training in many fields which require a small amount of chemistry—nursing, dietetics, forestry, basic engineering, comprehensive physical science for education majors, textiles, pharmacy, home economics, as well as liberal arts.

Many of these state schools offer several short courses in each branch of chemistry.

Usually the short courses are deemed satisfactory and adequate for students other than premedical students. In general the premedical student is urged to take the short course in quantitative analysis and the regular year course in organic chemistry. Six of the 31 medical schools also advise or recommend the short course in physical chemistry.

While the minimum requirements in chemistry for entrance to medical school vary rather widely, few entering medical students present the minimum. The average is about 18 to 20 credit hours of chemistry. The medical schools in general view a short course in organic as inadequate.

The attitude of the teaching faculty—and hence inevitably, the caliber and effectiveness of the short courses taught—depends largely on the importance attached to these courses by the administration. If the teacher feels that no recognition will attend a good teaching job, that preparation for the short course is merely time lost from activities which might advance him professionally—then he feels that short courses are unsatisfactory, and with reason. When the load of short courses falls fairly evenly throughout the department as a regular part of the teaching load, the attitude and result seem to be entirely different.

Replies from 118 Colleges and Universities of over 3500 Student Enrollment

Short courses offered for special curricula	*Number of schools*			
Quantitative analysis	39			
Organic chemistry	64			
Physical chemistry	57			
No short courses offered	30			
Does the short course cover essentially the same material as the regular long course?	Yes	18	No	63
Is the shortness achieved by				
(*a*) Limiting the number of topics treated?	15			
(*b*) Less rigorous consideration of topics?	4			
(*c*) Both means mentioned above?	65			
Does the short course contain special material, such as is pertinent to special groups which are not treated in the regular course?	Yes	32	No	43
Do the faculty feel that the short courses are somewhat unsatisfactory for the teacher?	Yes	37	No	44
Do the faculty feel that the short course is important, a challenge and an opportunity for the teacher?	Yes	49	No	30
Do the faculty teaching short courses have special training in allied medical fields such as zoology, biochemistry, or the like?	Yes	16	No	49

The short course in physical chemistry deserves a special word of comment. This course is usually sub-

The Quantitative Analysis Short Course

Hours of lecture and laboratory in the quantitative analysis short course offered by 26 colleges. All are one semester courses except as noted.

No. of Colleges	*Lecture*	*Lab.*	*No. of Colleges*	*Lecture*	*Lab.*
1	1	3	1	2	9
5	1	6	2	3	3
2	2	4	1	3	5
1	2	5	2	3	6
9	2	6	1	3	9
1	2	8			

One school offers a course of 1 lecture hour and 6 laboratory hours for two semesters.

stantially the same as the regular course, except that calculus is not used. This makes the course available not only to premedical students, but also to those majors in education who plan to teach science, and to those students majoring in chemistry for the B.A. degree, who otherwise would have no experience in physical chemistry.

The interest and degree of feeling on the part of the teaching faculty and the medical schools were apparent in the very prompt return of the questionnaires, accompanied in most cases by rather extensive comment and letters of observation. These comments have been mimeographed and may be obtained from the author on request.

ACKNOWLEDGMENT

The author is grateful to the 145 collaborators who made this compilation possible.

The Organic Chemistry Short Course

Hours of lecture and laboratory in the organic chemistry short course offered by 45 colleges.

No. of Colleges	*Lecture*	*Lab.*	*No. of Colleges*	*Lecture*	*Lab.*
One-Semester Courses			*One-Quarter Courses*		
2	2	3	1	3	0
1	2	4	1	4	3
1	2	6	*Two-Quarter Courses*		
11	3	3	2	3	3
6	3	4	1	3	6
8	3	6	*Two-Semester Courses*		
2	4	3	1	2	3
1	4	6	1	2	4
1	5	0	1	3	3
1	5	6	1	3	4
1	6	3			
1	6	6			

The Physical Chemistry Short Course

Hours of lecture and laboratory in the physical chemistry short course offered by 35 colleges.

No. of Colleges	*Lecture*	*Lab.*	*No. of Colleges*	*Lecture*	*Lab.*
One-Semester Courses			*One-Quarter Courses*		
3	2	3	1	5	0
10	3	0	*Two-Quarter Courses*		
8	3	3	1	3	3
3	3	4	*Two-Semester Courses*		
3	3	6	1	2	0
1	4	0	1	3	4
1	4	1			
1	4	3			
1	5	0			

PREPARATION OF SOLUTIONS OF PHOSPHORUS IN CARBON DISULFIDE

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In general, the procedure for preparation of solutions of white phosphorus in carbon disulfide is hazardous and unsatisfactory. A method is presented here which obviates most of the dangers and leads to solutions of higher purity.

Usually instructions call for the cutting of sticks of white phosphorus under water. These pieces are then dried with absorbent toweling to remove the water and then placed in a beaker containing the desired amount of the solvent, carbon disulfide.

If the pieces are too small, there is a serious danger of ignition upon drying. If the pieces are too large, solution is slow, and in either case the solution is contaminated with the oxides of phosphorus from the surface of the sticks, and water that was not removed during drying.

It is suggested that solutions be prepared in this fashion: Place the carbon disulfide in a separatory funnel, choosing such a size that the funnel will be less than half-full. Then cut the sticks of phosphorus under water into small pieces. These pieces are transferred *without* drying to the separatory funnel and 5 ml. to 10 ml. of water for every 200 ml. of carbon disulfide are added. This additional water may be necessary to break the emulsion formed when the CS_2/H_2O ratio is too large. The separatory funnel is shaken to dissolve the phosphorus (care being taken to relieve the pressure built up). A relatively pure solution of phosphorus in carbon disulfide can then be drawn off, since the water and carbon disulfide are immiscible and the surface oxides of phosphorus remain at the interface.

PYROLYSIS AND NAVAL STORES[1]

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THE pyrolytic processing of wood for tar and charcoal, both among the most essential commodities for a developmental technique, can be attributed to the earliest fire tamers. It is likely that the use of tarry products in combustion preceded that of charcoal, admitting that tars were certainly noticed by the cave man, the great user of fire-brands and the like for light and heat. Tars drip from torches or stream from burning resinous masses in the form of a dark fluid which hardens, upon cooling, to a brittle solid. Long eras must have elapsed before the practical importance of such a by-product was appreciated, while its only detected peculiarity was perhaps the high imflammability when blown as a powder above a flame.

Systematic and controlled fire treatment of wood was first attempted after the deduction that wood tars had many similarities with the natural bitumen, many uses for which were already established. Then the endeavor became more opportune, tars, pitches, and their compounds were manufactured and applied instead of bituminous matters, and eventually new uses were found for the wood products, stimulating the new industry. From deciduous woods charcoal was in time obtained.

BITUMEN

Solid asphalts, viscid malthas, oils, and combustible gases were encountered and used in many localities of the ancient world, Mesopotamia leading in abundance and in the awareness of their value. Peoples of Babylonia, short of wood, used dried asphalt to feed their fires, and lacking limestone for lime, cemented their bricks in construction with asphaltic mortar (*1*). Thus was the construction of the Tower of Babel attempted. To their bituminous cement was attributed the unyielding strength of the Walls of Babylon, still standing firm long after the city grounds were reduced to farming (*2*). Bitumen was also the bond in brick and stone pavements, even far from Asia, as in a temple in Greece (*3*); in the latter evidently for water insulation. The water-tight passage of Semiramis on the bed of the river Euphrates is a typical example of that kind of application.

More widespread and of a very ancient origin was the application of asphaltic compounds for protection of wooden ships, structural members, and other objects, (*4*), as this can be construed from the account about Noah's Ark and Moses' basket. Lifesize figures of bituminized wood were found perfectly preserved in the burial chamber of Tutankhamen.

Large shipments of black blocks fished from the Lake Asphaltites, the Dead Sea, and marketed as Yiddish or Syrian asphalt, were sent to Egyptian mortuaries (*5*), were eventually called "Pissa Tymon" in Greek, "Pix Tumens" in Latin, meaning tar or pitch of tombs, from which the term "bitumen" derived. This distinction became a necessity after the pine tars and pitches were found to be adequate substitutes for bitumen—even better than them in most cases. Only for embalming was bitumen found best qualified.

The diverse and extensive applications of the bituminous materials speak for a systematic processing of the raw asphalts to make them conform to given specifications for each particular application. The asphalts destined for combustion must be dry, *i. e.*, solidified, their light oils expelled, in order to promote burning and give controllable fires without the danger of flames "jumping out and burning the house" (*6*). The cements must be plastic enough for application, at the same time stiff to resist shear while being applied, their rate of hardening corresponding to the rate of added structural load. With materials so processed, strong and lasting brick-works were built, in the ruins of which the asphaltic joints are often found still intact and stronger than the brick. No brick can be detached with out breaking (*7*).

The paint for doors in Babylon could never have been of a black and sticky natural asphalt; in reality it is characterized as "hypekauma," a term denoting "product of underneath firing," *i. e.*, a distillate. Also, distillates prepared by pyrolysis of bitumen must have been the embalming liquids.

That a primitive form of fractionation was practiced is further suggested by the description of the oils known as naphthas, although considered by the ancients to be natural oils associated with nonsolid bitumen. Two kinds of naphthas were known, the white or real of the color of sulfur, and the dark or black (*8*). The former, described as "an extract draining from bitumen," was of restricted use due to its inflammability, attracting fire from a distance (*9*) and burning with long flames. The black variety had served as lamp-oil. Others considered the white naphtha with its astonishing qualities as a water (liquid) poisoned by inflammable solutes (*10*), while in reality it was just a light distillate of petroleum with low flash point, the black kind being a heavier fraction. However, the possibility of the white naphthas being turpentine is not excluded, because of the similarity of qualities and the fact that turpentine is still popularly called "nephti" in the countries surrounding the Mediterranean Sea.

Among the races living in or around the asphalt re-

[1] Presented at the 121st Meeting of the American Chemical Society, Buffalo, March, 1952.

gions of Asia were the Syrians—merchants, artful, and especially great fire workers. To them, as the best qualified by concurrence of circumstances, must be attributed the industrial development and compounding of the raw material still known as Syrian asphalt, and also the treatment of wood by the thermal methods used for bitumen. Thus an important new industry was initiated when wood tars and related products were found to be advantageous substitutes for most of the bituminous compounds, the raw material for which had to be collected in bloody fights by warriors using both arrow and axe. It is likely that the center of asphalt processing shifted later to Egypt, the country of origin of the chemical industries; the reports are that most of the bitumen from the Dead Sea was exported (perhaps crude) to Egypt at great profit. Egyptians dominated Syria for a while and had the opportunity to learn many Syrian arts.

WOOD TARS

The dawn of history finds the Syrians subjecting wood and resin from terebinth (*Pistacia Terebinthus*) to pyrolysis for tar, pitch, and terebinth oil (turpentine). From Phoenician cedar or juniper they were manufacturing a tar and the pyroligneous liquor known as cedria or cedrion (*11*). The wood and resin of Alepo pine were also worked by them. A light distillate, pine oil, was extracted from cones, probably the same as our modern solvent by that name, prepared from cones and needles of *Pinus Palustrus*. The heavy residue of cone distillation, the cona (*12*), was considered an inferior quality pissa. For some authors cona is synonymous with pitch.

The industry spread westward and found root in areas of coniferous tree growth, with working centers on the islands of Rhodes and Euboea, the mountain Ida (the most esteemed kind), the Macedonian Pierria of the Greek mainland, some north Italian areas, and the Galatian Vienna.

The all-around importance of tars, pitches, etc., in ancient life may be judged by the fact that Jacob included tar among the presents sent to the great man of Egypt—his own lost son (*13*)—and later by the Seleucidae of Syria contributing to the Rhodians loads of tar and resin along with other basically important materials (lumber, iron, etc.) for the reconstruction of their cities demolished by the historical earthquake (*14*).

Fortunately, the commonest process adopted for destructive distillation of resinous wood is recorded by Theophrastus in his "Historia Plantarum" (*15*). A technical interpretation of the method based on the Greek text and other available references is presented here.

The wood of wild pine (*Pinus Silvestris*), which contains abundant and very pure tar, was preferred as raw material. Mature and older trees were selected due to the notion that there is very little resin, if any, in the young ones; in them the sap is all consumed to promote upward growth and cone bearing. A tree has to reach full height before its sap goes to thicken trunk and roots, part of the sap being converted simultaneously into resin, a secretion pocketed in the wood (*16*). Trees most exposed to the sunshine and northern winds —meaning those from sparsely wooded areas—were preferable. This is in agreement with the fact that in dense forests trees grow tall and thin, and therefore are poor in resin. Exposure to the north winds should yield drier wood with smaller production of watery distillates; in Greece south winds carry the rain. Generally, the yield of the best quality tar was higher following mild winters. In Macedonia the male pines (those not bearing fruit) were preferred for tar and only those female ones which were found with resin-bearing roots (*17*). The selected trees were felled and their trunks cut in equal lengths, because of the kiln design, and split up for the pyrolysis.

To start a kiln, a circular piece of ground 180 cubits in circumference, or about 80 feet diameter, was cleaned and worked smooth and slanted toward its center, from which a duct was opened with a gradient for conveyance of the liquids of pyrolysis to the tar pit. This was dug at a distance from the pile sufficient to allow the liquids to cool before entering the pit (about 20 feet). The well-smoothened duct was slated even with the ground,[2] obviously for protection against stoppages liable to be caused by pieces of wood falling in during the pyrolysis, as well as against contamination of the product. Then the building of the pile proceeded with a kind of tunnel left in the lowest courses intended for the starting of the fire. The stacking of the wood was similar to that employed in the making of charcoal, except for the hole.[3] The pieces were arranged upright so that the kiln increased in height with the piling. The work was completed when a height of 50 to 60 cubits (70 to 85 feet) was reached. However, these dimensions sound absurd to us; every pitch kiln was then supposed to be built as high as the Colossus of Rhodes (80 feet), one of the Seven Wonders, with a statically impossible ratio of base diameter to height roughly 1:1. In view of Theophrastus' accuracy and excluding a copyist's mistake as rather incredible in this case, the only alternative is to assume that the measurements were taken correspondingly: the height was measured circularly just like the circumference, *i. e.*, from one end of the base diameter up and around the pile to the other end of the diameter. If, instead of circumference, diameter were given for the base, then the height should be taken as linear, in our familiar way. From the above consideration the linear height of piles was between 23 and 28 feet. For modern piles the ratio falls between 2:1 and 2:1.5. Furthermore, modern kilns would logically correspond in dimensions to the ancient piles. As for the dimensions of 100 cubits for both circumference and height, specified in the text for the pyrolysis of wood rich in pitch, these can only refer to stationary masonry kilns of cylindrical shape, not higher

[2] This is the meaning of the verb *edafizo*.

[3] This hole (*18*) is the flue left in the middle of the charcoal pile after the posts around which the wood was heaped-up for charring were pulled out.

than 25 feet (the base counted also in measuring), and similar to the modern vertical steel kiln for the same purpose.

Forest litter and lute were applied to the surface of the kiln to keep flames from leaping out, after which the fire was started inside the tunnel. Then the mouth of the tunnel was sealed with the same materials, only the duct channel remaining open for admission of an amount of air just necessary to maintain the temperature not much above the degree of pyrolysis by slow combustion. Ladders were handy for checking any flame emerging from the luted pile. The process lasted for two days and nights, the operators offering sacrifices and making merry with wishes for good and plentiful tar. Before sunset on the second day the fire was out.

Eventually the kiln was built stationary with spherical dome, all of brick, the next development being the outside-heated unit for the sweating-out of tar. Such probably were the Byzantine tar-kilns of the sixth century, mentioned in the Maspero Papyrus (*19*), perhaps not much different from the kiln known as American, forerunner of the horizontal retorts. It must be noted here that in a locality (Spathareoi) of the Island Samos, where modern methods have not as yet penetrated, they extract tar from strips of wood loaded in a sheet iron container, provided with an opening at the bottom for starting the fire, and letting the tar so formed drip into pits dug in the ground.

From hearsay, Theophrastus knew of another method used in Syria for treatment of resinous woods. A heater, an instrument specially fashioned, was applied directly to the trunk and transfered from tree to tree, sweating-out tar instead of resin, as by incision. Does the method now in use for obtaining Peru balsam descend from Syria?

As for the empyreumatic distillate, the cedria, this was used in the preservation of dead bodies from all corruption (*20*). The dense tarry product was either washed for finished tar of improved quality or treated again with fire for other products. The washing was done with a solution of potash prepared by filtering water through ashes of grapevine branches. The alkaline solution was added warm to the slightly heated tar in earthenware containers and in open air.[4] Following thorough mixing and two hours standing, the supernatant liquid was decanted and the washing repeated with fresh warm solution of potash three times a day for three days; after final decantation any solution still remaining on the surface of the tar was removed with a sponge.

Tars are brown to black. Samples newly prepared from pine in an iron retort were dark yellow, darkening to brown on exposure but without sedimentation. In thin layers tars are translucent, an advantage for some of their applications. However, they were mainly valued as raw materials for production of pitches and other naval stores.

[4] The text reads "in sunshine," in reality meaning "not under roof," because of the fire hazard (*21*).

PITCHES AND OILS

By lengthy boiling of the tars in copper stills, the tar serum or tar oil (light oil), free of watery products, was distilled off and the pitch remained,[5] the "palimpissa," meaning "again tar" or "tar also." Earthenware stills were also in use, as in the preparation of the medical tar-oil (*22*), undoubtedly long-neck retorts like those of the third century Byzantine alchemists. The mouth of the retort was plugged with clean wool in which the oil condensed, to be squeezed out in a receiver. Probably the wool was wick-shaped and extended into the container, thus functioning as condenser; the squeezing was effected on the extending part of the wick.

Pot boilers similar to the food-cooking vessels of wood or skin of primitive Americans were also used in making pitch from resinous materials. They consisted of strong oak troughs, recipients of the resin, into which red-hot stones were thrown (*23*).

By a kind of steam distillation of resins, turpentine was separated from the pitch, *i. e.*, the rosin or colophony, named after Kolophon, the Lydian city. Turpentine owes its name to terebinth, as previously noted, being probably first produced from its resin.[6]

Resins were extruded by tapping trees (*24*), the operation not having changed much since antiquity, and collected and kept in leather bags for thickening (*25*). Expelling the light oils from resinous matters was considered a drying operation, leaving behind viscous, solid, or entirely dry products. The soft types were known as "voskas," denoting stomach or belly, either due to their consistency or to the fact that they were the middle fractions—middling in a distillation in which tar oils were the heads and pitch the tails.

Pitches were substituted for natural bitumen in many applications, in consideration of their lighter color and their consistency, usually thinner than the commonly known natural pitches (*26*). Due to the similarity, varieties of asphalts (solid) were called pissasphalts (*27*). As late as the tenth century A.D. the asphalt of Zakynthos was understood to be liquid pitch of pine produced without action of heat (*28*). Latins called pix the Macedonian pitch, and bitumen, the Babylonian asphalt (*29*). However, bitumens are often called pitches.

According to the ancient specifications, a pitch of good quality should be glossy, of pleasant smell and sweetish taste; when heated it should not jump (throw out droplets), nor squeek, nor bubble; ignited it should give a dense smoke, such density being a sure criterion of quality; the denser the smoke, the better the pitch. The black color characterizes the pitches and the expression pitch-black was used by Homer, who considered nothing blacker than pitch (asphaltic). However pitches are not absolutely opaque; in fact, like the tars,

[5] Additional vinegar, according to Latin information (*20*), is objectionable, the liquid being acid (or even neutral) and brought to boil in copper vessels.

[6] Steam distillation was practiced long before the Arab Avicenna popularized the water solutions of flower essences.

in thin layers they are translucent with a reddish hue, a property upon which some of their applications were based.

A singular type of pitch was the so-called "zopissa," consisting of scrapings from ships that had been smeared with a mixture of pine tar or pitch or resin and wax (*30*). These scrapings, a wonderful finding of human art, Pliny declares (*31*), are preferable for every application where tarry matters are needed because of combination with the sea salt.

USES

"If it weren't for pitch and resin nothing could float safely on water" (*32*); this is an axiom for the wooden-ship builder whose essential materials, besides the proper lumber, the flax and the hemp, are pitch and wax, the ship being sealed with them to be made tight (*33*). So, the products of resiniferous wood treatment were of prime importance and are still labeled naval stores: the pine pitch, tar, and oil, the resins and their oils or spirts, etc.

The ship builder in northern countries where temperature fluctuations between extremes and higher humidity prevail, especially by the sea, thought of smearing his wooden shed with the materials used on the ship (*34*). In southern countries pitch compounds were handy for wood protection, instead of the bitumen of the Babylonian pattern. For that purpose pitch, resin, and wax were mixed (*35*), probably also with a drying oil. Pitch alone or in various mixtures with wax, animal glue, oils, and ochre (for body and color) was used in Greece for coating the lumber on roofs of other wooden articles (*36*). The Ephesians painted their wooden statue of Artemis with nard oil (*37*). Cedar light distillate or cedar-resin oil was applied in embalming by economy minded Egyptians (*38*) and also by the European Galatians for preservation and display of the heads of their most hated enemies and by the Belgians for the heads of their departed prominent men (*39*).

On roofs, tar and pitch compounds were also applied to protect clay tiles (*40*) against both water soaking and vegetation growth. Common ancient tiles are usually very permeable and often soft-burnt.

Another important application of pitches, resins, and tars was in leak-proofing wine barrels, practiced in the countries watered by the river Padus (Po, Italy) and in Vienna, where both lumber and pitch were in abundance (*41*). There the pix wine was fermented and stored in wooden containers as large as a house, which were plentifully smeared inside with pitch. Also pottery intended to hold liquids was pitch-coated inside soon after it was taken from the kiln. Wine was exported to Egypt from Greece in tarred ceramic jars (*42*), this being verified by excavated potsherds still holding pitch (*43*). The leather bags of the ancients were treated with resins and resins are still among the finishing materials in tanning.

An uncommon application of pitches was in the dressing of masonry walls with marble slabs. In that case the pitch served as glue besides insulating the slab. It was poured hot to fill the space between the surface of the wall and the back of the slab (*44*). The slabs of luxurious marbles dressing the inside of Aghia Sofia in Constantinople were set with pitch, as can be detected by inspection of detached pieces.[7]

Because of the protective properties of the materials in question, they were used for anticorrosive treatment of metals, especially copper masterpieces (*45*). Pitch, coated on copper, gives a darker and softer hue to the metal. The application on iron was called by the Greeks "antipathia," meaning "suffering instead" and it was performed with religious rites. The antipathia paint was compounded by mixing white lead and terra alba (an extender) with liquid pitch. Pitch alone, applied hot, was the paint for the heads of iron spikes, pieces of machinery, and iron articles in general (*46*).

Tarred felt was manufactured for waterproofing long before A.D. 1870, when Swedes introduced the coating of thick paper with hot mixtures of pitch and pigments for roofing: namely, tar paper. It is reported (*47*) that the heavy copper nails decorating the private ship of Ieron, the Tyrant of Syracuse, were insulated from the wood by a lead leaf resting on cloth impregnated with pitch. On the same ship, the water storage was built with wooden boards lined with pitch-soaked cloth (obviously the boards formed a double wall and the cloth in between was felt, very well known to the ancients).

Pitches and resins had their uses in warfare, when pots filled with tarry mixtures (asphaltic also) were set on fire and thrown upon the enemy (*48*).

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(20) Pliny 16.21
(21) Geoponica 6.5.1 sq.
(22) Dioscurides, *op. cit.*, 1.72.3
(23) Pliny 16.22
(24) *Ibid.*, 16.23
(25) Theophrastus, *op. cit.*, 9.2.1

[7] Kodinos, an eye witness of the construction of the famous church, reports that a lime-oil mortar was the gluing material.

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SOME ACCIDENTS IN JAPANESE SCHOOL LABORATORIES

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GENERALLY speaking, accidents in chemical laboratories are not reported by school authorities. Hence the Ministry of Education of the Japanese Government does not have the total number and data of such accidents. However, the following are accounts, with comments, of some accidents which were reported by the press.

(1) August 15, 1944. The report says that during an experiment an assistant in the Department of Chemistry of Tokyo University died from an explosion. The explosion was probably caused by organic matter in a liquid air trap. Liquid nitrogen is preferable to liquid air as a cooling agent.

(2) August 8, 1950. In the Aomori-prefectural Hatchinoe high school more than ten students were injured in the arms by test tube explosions. Following instructions in their textbooks, they carried out an experiment to: "Ascertain the fact that oxygen is generated by the addition of concentrated sulfuric acid to potassium permanganate and heating." This explosion was probably caused by the formation of Mn_2O_7, an unstable, dark brown, oily liquid. It decomposes explosively into MnO_2 and O_2 at about 70°C. Safety depends upon a suitable excess of H_2SO_4, which dissolves Mn_2O_7. The mixing ratio of 0.2 g. of $KMnO_4$, to 3—3.5 g. of concentrated H_2SO_4 is suitable for high-school students. This case shows a lack of knowledge and caution, and, moreover, a lack of preliminary experiment.

(3) June 27, 1952. In the primary school in Niigata an explosion occurred when the teacher ground a mixture of red phosphorus and glass powder, in order to make a match box. Forty-three people were injured. It was reported that the explosion was caused by $KClO_3$ which had been left in the mortar. The teacher had used the same mortar in the previous lesson to make a match head by mixing MnO_2, S, and $KClO_3$. The heat of friction caused the red phosporus to ignite explosively with the oxygen of the potassium chlorate. This case shows extreme carelessness. Also, large amounts of chemicals were probably used. It is essential to remember that only small quantities of hazardous chemicals should be used.

A few likely sources of accidents are as follows:

A Detonating Gas. Many accidents are caused in primary and junior high schools by explosions of detonating gas. Care must be taken when hydrogen or a mixture of hydrogen and oxygen is ignited.

Kipp Generator. Hydrogen sulfide is usually generated in a Kipp's apparatus. When a Kipp generator is filled with sticks of FeS, one must be careful not to fill the lower compartment, or to use small sticks or dust of FeS.

Poison of Hydrogen Sulfide. Hydrogen sulfide is a common reagent in chemical laboratories. Its toxicity is apt to be forgotten. The author was reminded of it when a cat was an accidental victim!

Ammonia. In the summer re-education course for junior high-school teachers, a teacher opened a bottle of ammonium hydroxide (28 percent solution), the contents of the bottle burst out, and four teachers were drenched. When a bottle of ammonium hydroxide is opened in warm weather, care must be taken.

Fires. After the war several schools in Japan were burnt down from various causes: (*a*) ignition of soybean oil, (*b*) defective electric heaters, (*c*) leakage of water onto metallic sodium, (*d*) electrical ignition of oil on the floor of a generator room.

THE RELATION OF HIGH-SCHOOL PREPARATION TO COLLEGE CHEMISTRY GRADES[1]

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THE GRADES made by students in beginning college chemistry courses have been, and are, a matter of concern to the authors and their colleagues. It would seem, after comparing chemistry grades with those made in the other subjects at Southern Illinois University, that here, at least, chemistry is more difficult or that a higher level of achievement is required than in many other courses. While many factors are involved in proficiency in college chemistry, previous training and performance in certain high-school courses should be important. Pertinent information concerning such courses can be obtained from high-school records which in this case were made available by the University Registrar.

Work of this general type is not new. Scofield (*1*) in 1930 concluded that good high-school mathematics grades, especially when combined with good high-school chemistry grades, are better than good placement examination grades for picking good students. Herrmann (*2*) in 1931, Steiner (*3*) in 1932, and Clark (*4*) in 1938 showed that, in general, high-school chemistry is advantageous to students who enroll in beginning college chemistry while West (*5*) in 1932 concluded that factors such as intelligence are more important than specific high-school training.

The above studies, while informative and interesting, did not satisfy completely the questions the authors had in mind. It seems that high-school physics, as well as chemistry and mathematics, might have some bearing on this question. Then, too, this problem chiefly concerns students from Southern Illinois with its large but widely dispersed population. Many high schools are involved and their courses vary in content and thoroughness. This makes this investigation specific and local and any conclusions will be general only in so far as the same conditions pertain to other areas.

The purpose of this study, therefore, was to examine the high-school records of 696 students who enrolled in the beginning college chemistry course, described later, during the years 1947 to 1950 inclusive. The grades these students made in high-school chemistry, physics, and mathematics were determined and an attempt was made to compare those data with the grades these students made in the first term of the beginning college chemistry course taken by all students who plan to take one year or more of college chemistry.

This college chemistry course is one based upon the assumption that the students had no high-school chemistry. It starts with fundamentals of atomic structure, nomenclature, equations, etc., and goes through the study of the halogens. The course is fairly thorough and has three hours of lecture and four hours of laboratory per week, for twelve weeks. Some of the more theoretical and mathematical concepts such as gram molecular weight determinations, equilibrium constants, colligative properties of solutions, extended studies of the theories of electrolytes, etc., are not included in this course but are delayed until the second term.

The major subjects of the students include chemistry, zoology, botany, pre-medicine, pre-dentistry, pre-engineering, etc., and no differentiation is made on the basis of having or not having had high-school chemistry, physics, or mathematics. These students decided in high school, or prior to entering college, on a field of training that makes good grades in chemistry important for success. It seems reasonable to assume, therefore, that they had some incentive for the study of chemistry prior to enrollment in the college chemistry course.

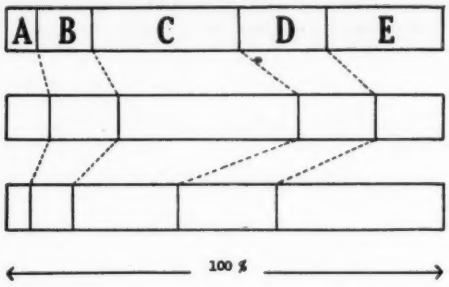
This investigation definitely does not include students who took college chemistry only to satisfy a requirement in physical science for graduation. Such students take an entirely different course from the one mentioned above.

The study resolved itself into two parts. First, an attempt was made to determine the effect of courses in high-school chemistry, physics, and mathematics (merely, whether a student had such courses or not) on college chemistry grades. Second, an attempt was made to correlate proficiency in high-school chemistry, physics, and mathematics with proficiency in the college chemistry course previously described.

It should be emphasized that the data presented below apply to the specific group of students taking the specific college chemistry course previously described. No data were obtained on the students who took other kinds of beginning college chemistry courses.

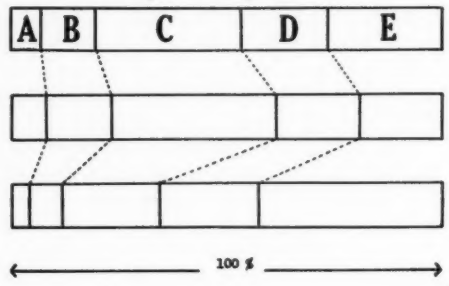
The results of the first part of this study are shown in Figures 1 and 2. Included in these figures, for purposes of comparison, is the graph of the grades made in this college chemistry course by all of the students in the group. The disturbing feature of this graph is the large per cent of D and E grades and the low per cent of A, B, and C grades. The instructors of

[1] Presented before the Chemistry Section of the Illinois State Academy of Science, May 4, 1951.



	No. of Cases
College chemistry grade distribution	696
Had h.-s. chem., physics, and math.	346
Without one or more of h.-s chem., physics, or math.	342

Figure 1



	No. of Cases
College chem. grade distribution	696
Had h.-s. chemistry	468
Had no h.-s. chemistry	228

Figure 2

this course feel that their teaching ability is not unreasonably low and that their standards of achievement are not unreasonably high. The important question is, what factors account for such results? It may be stated here that this study does not completely answer this question.

The data, from which the graphs in Figures 1 and 2 were made, were analyzed statistically and the graphs show real differences at all of the grade levels. Graph 2, Figure 1, is for the group that had at least some high-school chemistry, physics, and mathematics. This group had the lowest per cent of D and E grades, and the highest per cent of the other three college grades. Graph 3, Figure 1, is for the group that did not have the combination of high-school chemistry, physics, and mathematics. They lacked one or more of these three high-school courses. Their poorer record in college chemistry is apparent from the graph. Undoubtedly, there are several reasons for the differences in the graphs 2 and 3. Native intelligence, interest in science and mathematics, and the willingness to work probably are involved.

Graphs 2 and 3 in Figure 2 show real differences at all of the grade levels between the groups that had and those that did not have high-school chemistry. The group that had high-school chemistry shows a marked superiority with respect to college chemistry. Graph 2 owes some of its characteristics to the fact that this group contains many students whose records produced graph 2, Figure 1. Likewise, the students involved in graph 3, Figure 2 are part of those included in graph 3, Figure 1.

The second part of this study did not lead to definite results. The attempt to correlate proficiency in high-school chemistry, physics, and mathematics with college chemistry grades did not produce real differences between the various groups. It is felt that certain observed trends would lead to real differences if enough cases were observed. For example, it appears that as the amount of high-school mathematics taken increases, the proficiency in college chemistry also increases. This, perhaps, is to be expected but, again, other factors are probably involved.

Since the data were available, the graphs in Figure 3 were included to show the high-school chemistry, physics, and mathematics grade distributions for the students involved in this study. Graph 1 for the college chemistry grades is included for comparison.

The following observations may be made concerning the students and the college chemistry course involved in this study.

(1) Students who had a combination of high-school chemistry, physics, and mathematics made the best records in this beginning college chemistry course. It was not possible to determine how much of this was due to the high-school courses themselves and how much to other factors.

(2) Students who had high-school chemistry, irre-

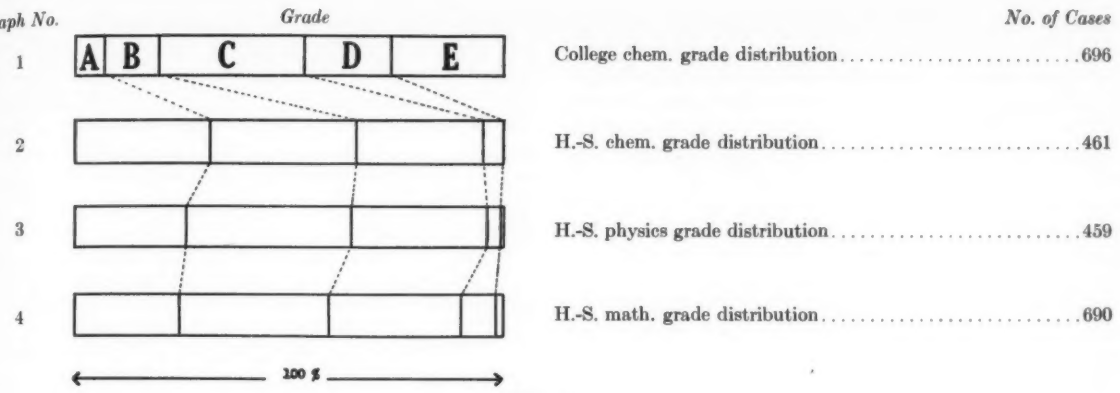


Figure 3

spective of other courses, made better records in this college chemistry course than those who did not have high-school chemistry.

(3) The most common grade made by these students in high-school chemistry, physics, and mathematics was a B.

LITERATURE CITED

(1) Scofield, M. B., J. Chem. Educ., **7**, 117–26 (1930).
(2) Herrmann, G. A., *ibid.*, **8**, 1376–85 (1931).
(3) Steiner, L. E., *ibid.*, **9**, 530–37 (1932).
(4) Clark, P. E., *ibid.*, **15**, 285–9 (1938).
(5) West, G. A., *School Sci. and Math.*, **32**, 911–3 (1932).

FRANZ HEIN

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University of Cincinnati, Cincinnati, Ohio

Franz Hein, eminent chemist and teacher, was born at Grötzingen (Baden) on June 30, 1892. He early developed a liking for nature and natural history, together with an interest in technical matters. These interests grew logically into a choice of chemistry as a career, a choice that was particularly favored by the family's removal to Leipzig, where his father, an artist, took a teaching post. At this time Leipzig was one of the leading centers of chemical education, and the influence of Wilhelm Ostwald was still quite potent. Hein studied at the University of Leipzig from 1912 to 1917 under Arthur Hantzsch (1857–1935) who had guided the doctoral dissertation of Alfred Werner (1866–1919), the founder of the coordination theory.[1] Hantzsch's temperamental enthusiasm for chemistry and his real scientific competence made a powerful impression on the students. His inorganic colleague was Konrad Schaefer (1874–1922) who as pupil of H. Ley had come to appreciate thoroughly the value of absorption spectra as a key to constitutional problems; and he, like Hantzsch, used the methods inaugurated by Hartley and Baly for the qualitative determination of absorption curves, particularly in the ultraviolet.[2] Consequently, Hein's dissertation, prepared under Schaefer's and Hantzsch's direction, dealt with problems whose solution demanded not only preparative skill but also familiarity with spectral methods. The thesis dealt with optical studies of bismuth compounds and triphenylmethane derivatives, topics which involved intensive occupation with both inorganic and organic chemistry.

Even then, Hein began to appreciate the importance of the boundary region between the organic and inorganic fields; he realized that studies of the organo-

[1] Pfeiffer, P., J. Chem. Educ., **5**, 1090 (1928).

[2] For an outline of Schaefer's career see Hein, F., *Z. angew. Chem.*, **35**, 444 (1922).

metallic compounds and the chemistry of complexes would supply valuable insights into the chemical behavior of metals. With these points of view, he began to search for organic derivatives of chromium, a search that was rewarded by the discovery of the phenylchromium compounds. He thus opened up a territory that has not yet been completely explored. This work was used as the basis for his habilitation as Privatdozent in chemistry at Leipzig in 1921. In 1923 he was appointed assistant professor in inorganic chemistry at this university, where he had already served as assistant. About this time, he proved that substitution of organic radicals for hydroxyl in metal hydroxides generally results in bases, R_n-$_1M''OH$, that are stronger than the parent $M(OH)_n$. However, the nature of the metal is of decisive importance, and really strong bases were found only when chromium and thallium hydroxides were used.

At the same time it was shown that the solution melts of alkali metal alkyls in zinc ethyl and related substances are good conductors of electric current; this finding was used to demonstrate the salt nature of even the simple alkali metal alkyls. A study of the electrolysis of these compounds showed that the Faraday laws hold in these systems. Furthermore, the free alkyl radicals formed at the anode were found to be highly active; they dissolve metals such as zinc, lead, aluminum, cadium, etc., with production of the corresponding metal alkyl. It was also shown that metal alkyls, *e. g.*, zinc ethyl, zinc propyl, and aluminum ethyl, provide dissociating solvents not only for alkali metal alkyls but also for electrolytes as strong as quaternary ammonium iodides.

These studies, like those continuing the investigation of chromium organic compounds, led, in constantly increasing measure, into the region of complex chemistry, which consequently came more and more into the foreground of his interest. One of the fruits of this part of his research efforts was the clarification of the relations between complex constitution and the capacity to form metallo-organic compounds. A new method was developed for the preparation of symmetrical organo mercury compounds in highly pure form, and also a complex chemical synthesis of α,α'-dipyridyl from pyridine and sublimed ferric chloride.

Other studies dealt with the reactions between chromous halides and ethers, which resulted in the formation of the chromium dihalogen alcoholates, which form complexes very readily. Spatial (tetrahedral) coordination was demonstrated in the silver oxyquinoline complexes $[Ag(oxine)_2]$ X by resolving them into optically active components. Triaminotoluene was prepared and its complex chemical behavior was studied. The reactions between organo metallic bases and iron pentacarbonyl and its hydrogen derivative were investigated and, for the first time, a series of well-defined organo metallo iron tetracarbonyls was obtained. The study of the complex chemical behavior of chromous iodide finally resulted in the preparation of pure hydrazine complexes $[Cr(N_2H_4)_n]\,I_2$. In addition, the α,α'-dipyridyl complex salt $[Cr(dip)_3]I_2$ was isolated; its characteristic behavior inspired further studies which led to the preparation, for the first time, of definite compounds of univalent chromium, *e. g.*, $[Cr(dip)_3]ClO_4$.

Among his other studies of metallo organics, special mention should be made of the findings regarding lead compounds. In particular, he arrived at an explanation of the autoxidation of tricyclohexyl lead. In the presence of halogenated hydrocarbons such as carbon tetrachloride and tetrabromide the latter take a decisive part in the reaction, which may then even become explosive. The phenyl derivatives of molybdenum and tungsten were described; their stability is far less than that of the corresponding chromium compounds, and they have not been so clearly defined. The thermal decomposition of toluene in the presence of mercury led, via the formation of benzyl mercury, to the proof of the intermediate production of benzyl radicals.

In the inorganic and analytical fields, he has investigated, among other topics, the behavior of silver permanganate, which because of marked deformation shows great reactivity with molecular hydrogen at room temperature. This characteristic has been made the basis of a new method of determining hydrogen. Mention should also be made of the equilibrium studies of chromium halides. Chromous iodide was obtained in well-defined form for the first time and thus could be characterized.

In the field of colloids he has contributed to the chemistry of molybdenum blue and has prepared sols and gels of chromium oxyhydrate from the ammine complexes of chromium dihalogen ethylates.

His wide experience in the field of coordination compounds made it almost inevitable that he would compose a text dealing with this important and extensive field. His "Chemische Koordinationslehre" was published in 1950; this book of 683 pages has had very favorable reviews. Together with G. Bähr, he furnished the article "Wasserstoff" and "Wasser" for the Fresenius and Jander "Handbuch der analytischen Chemie." Sections on atomic weight, periodic system, systematics of inorganic chemistry, coordination theory, and isomerism of inorganic compounds, by Proskauer and Hein, were included in the Drucker-Proskauer "Taschenbuch."

Since 1942 Professor Hein has occupied the chair of inorganic chemistry at the University of Jena. He declined an offer in 1949 to return to the University of Leipzig, his *alma mater*. In 1951 he was elected to the Saxony Academy of Sciences at Leipzig.

THE IDENTIFICATION OF ESTERS OF PHENOLS

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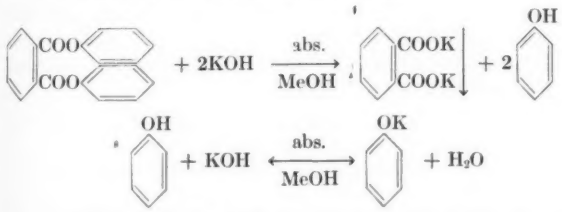
THE usual chemical methods for the identification of esters involve purification of the ester, determination of its physical constants, and hydrolysis of the ester by some method and subsequent isolation of the acid or alcohol fraction or suitable derivatives of these. The common methods used to isolate the acid or alcohol fraction depend on such physical properties as solubility and volatility. By and large, however, these methods are tedious and give poor separations.

The present modification is designed to better characterize esters of phenols and is based upon the observations that the potassium salts of phenols are soluble in absolute methanol while the potassium salts of organic acids are relatively insoluble in absolute methanol.

During the course of an investigation designed to establish the identity of an ester, which was later shown to be diphenyl phthalate, it was noticed that in the course of a saponification by means of absolute methanolic potassium hydroxide a dense white precipitate was formed.

This material was filtered out, washed with absolute methyl alcohol, dissolved in water, and acidified with concentrated hydrochloric acid. A white crystalline precipitate formed which was filtered out and recrystallized from boiling water.

The material was dried and identified as phthalic acid. The reactions involved are as follows:



It will be seen from the equations that because of the acidity of phenol, the potassium salt was formed in the saponification. More important, however, is the fact that it was soluble in absolute methanol whereas potassium phthalate was insoluble and precipitated out. This is of course due to the fact that potassium phthalate is stable and largely ionic in character; therefore, tending to be insoluble in absolute methanol.

In accordance with this observation and its implication it was decided to test the solubility of the potassium salts of other phenols in absolute methanol. The following phenols were tested by adding each individually to a half normal solution of potassium hydroxide in absolute methanol: phenol, catechol, resorcinol, β-naphthol, *o*-cresol, *m*-cresol, *p*-cresol, guaiacol, thymol, α-naphthol, pyrogallol, hydroquinone, and eugenol. In each case the phenol dissolved with the evolution of an appreciable quantity of heat and remained dissolved even on cooling. These results indicate that the potassium salts of phenols are soluble in absolute methyl alcohol.

The method therefore consists of the following:

(1) The ester is identified as an ester by means of the hydroxamic acid test[2] and other qualitative ester tests.[3]

(2) The ester is identified as an ester of a phenol by means of the Davidson indicator method.[4, 5]

(3) The physical and chemical constants, (m. p., b. p., saponification equivalent, etc.) are determined and a list of the possibilities is collected from the literature.

(4) The ester is saponified by means of absolute methanolic potassium hydroxide.[6] This method will generally be applicable since esters of phenols are rather readily hydrolyzed.

(5) The resulting precipitate of the potassium salt of the acid moiety is filtered and washed with absolute alcohol. The acid is isolated and identified as such or by means of a derivative.

If the compound is suspected to be an ester of a simple acid as acetic, it is recommended that absolute *n*-propyl or isopropyl alcohol be substituted for the methyl alcohol, since the potassium salts formed may be too soluble in the latter.

It is important that conditions be kept as nearly anhydrous as possible. It is firmly believed that by observing the precautions and conditions outlined the identification of esters of phenols would be accomplished with less difficulty and greater certainty.

[1] Present address: Central Testing Laboratory, Department of Purchase, City of New York, New York.

[2] DAVIDSON, D., J. CHEM. EDUC., **17**, 81 (1940).

[3] SHRINER, R. L., AND R. C. FUSON, "Systematic Identification of Organic Compounds," 3rd ed., John Wiley & Sons .Inc. New York, **1948**, pp. 61, 129.

[4] DAVIDSON, D., J. CHEM. EDUC., **19**, 154, 221, 532 (1942).

[5] "More Acids and Bases," J. CHEM. EDUC., Easton, Pa., **1944**.

[6] It is recommended that the absolute alcoholic potassium hydroxide solution be made by dissolving c. p. potassium metal in absolute methanol, cooling, and adding water stoichiometrically. However, sufficient water should be added to convert completely all the potassium methylate to potassium hydroxide, care being taken to limit the excess of water to a minimum.

Proceedings of the

PACIFIC SOUTHWEST ASSOCIATION OF CHEMISTRY TEACHERS

MEETINGS OF THE SOUTHERN CALIFORNIA SECTION

The La Jolla Meeting, held at the Scripps Institution of Oceanography on September 29, 1951, featured a tour of the new aquarium building, the best equipped on the west coast and housing a fine collection of deep sea fish. The formal meeting consisted of a panel discussion on "The chemistry programs in high schools and junior colleges and their integration with college chemistry." Panel members, representing the different types of institutions, were:

Dudley H. Robinson, San Diego State College.
Finis A. Johnson, Carlsbad Junior College.
David Ferris, San Diego Junior College.
Louise Hines, Herbert Hoover High School.
Paul Farrar, Point Loma High School.

The discussion brought out the differences in the general educational objectives in the various institutions, and how these aims influence the type and content of the chemistry courses taught.

The luncheon, which followed the morning program at one of the La Jolla hotels, was presided over by Norris W. Rakestraw. Ross Baker gave an interesting talk on his extensive experience in chemistry teaching.

December Meeting, 1951. On Friday evening, December 7, the Pacific Southwest Association of Chemistry Teachers sponsored the principal speaker at the Group Meeting of the Southern California Section of the American Chemical Society. Dr. William G. Young, Dean of the Physical Sciences at U. C. L. A. spoke on "The professional training of chemists and the function of the A. C. S. Committee on Professional Training." The meeting was held on the campus of the University of California at Los Angeles.

The first PSACT *News Letter* was sent out to members in December of 1951 by the editor, Robert Isensee of San Diego State College. Also published the same month was the first Membership Directory by the Association's secretary, Dr. Blanche Bobbitt, Science Supervisor of the Los Angeles Secondary Schools.

An interesting plant trip, a tour of the Sanitary Engineering Laboratories of the Los Angeles Department of Water and Power, on Saturday morning, February 16, 1952, was the most popular PSACT event of the year. Eugene M. Discant, Research Chemist at the Laboratories, gave a special lecture on the "Laboratory control of water quality," illustrating his talk by a film on water purification. Mr. Discant conducted the tour through the chemistry, biology, and bacteriology departments of the laboratories, where PSACT members saw the latest instrumentation methods in use, and also substantial proof of the claim that the Los Angeles area has the most germ-free water sources in the nation.

The Educational Division Program in the Regional Meeting of the American Chemical Society held at San Diego State College on May 10, 1952, was sponsored by the Pacific Southwest Association of Chemistry Teachers. Papers on the program, arranged by Chairman Philip Gill, were the following:

Robert L. Pecsok, U. C. L. A., "Polarography and the Versene complexes."
Kenneth N. Trueblood, U. C. L. A., "Chromatographic adsorption affinity and molecular structure."
Albert W. Bellamy and Joseph M. Heslep, Chief and Chemist, respectively, of the Radiological Services of the California State Office of Civil Defense, "The recycling of fission products in the biotic cycle."

Installation of officers for 1952–53 for the Southern California Section of the PSACT took place at the evening meeting on May 23, 1952, at the Pasadena City College. Assuming offices were:

Frank L. Lambert, Occidental College, *Chairman*.
W. Roy Newsom, Whittier College, *Chairman-Elect*.
Mrs. Dorothy Craig, Marlborough High School, *Secretary*.
William D. Leech, Pasadena City College, *Treasurer*.

Guest speaker of the evening was Robert T. Merrick, Assistant Director of Research at Don Baxter, Inc., who gave an illustrated lecture on "Recent developments in plasma extenders."

The following appointments were made by Chairman Frank L. Lambert for 1952–53:

Sheila Murray Bauer, South Gate High School, Chairman of the Membership Committee.
Philip L. Bruce, Long Beach City College, Member, Program Committee.
Sister Agnes Ann, Immaculate Heart College, Chairman of the Publications Committee.

On Saturday, September 29, 1952, Glenn T. Seaborg, recent Nobel Prize Winner from the University of California's Radiation Laboratory, addressed a meeting of the Southern California Section of the PSACT, held on the Occidental College campus. Dr. Seaborg's talk was interesting and informal, highlighting various stages in the development of the chemistry of the transuranium elements.

At the luncheon, which followed the morning program, special tables were designated for "shop talk" on the teaching of the different fields of chemistry. Members indicated their choice of subject matter and

were assigned to tables with other members of similar interests. This was an experimental "mixer," devised to promote the exchange of ideas among teachers of the same subject matter courses on an informal level. It was successful enough to warrant repetition, and will be used from time to time at future meetings.

The new chemistry building at U. C. L. A. was the place of meeting of the Southern California Section of the PSACT on Saturday, November 15, 1952. The morning meeting consisted of a panel of speakers on "Laboratory experience in college chemistry." These were:

Norman Davidson, California Institute of Technology, on general chemistry laboratory.
Conway Pierce, Pomona College, on quantitative analysis.
Thomas Jacobs, U. C. L. A., on organic chemistry laboratory.

After the luncheon, Francis Blacet, Chairman of the Chemistry Department at U. C. L. A., gave an account of the department's graduate program. The rest of the afternoon was given over to faculty-conducted tours of the beautiful new chemistry building, just completed at a cost of approximately five million dollars.

A field trip took PSACT members to Brea, California, on February 14, 1953, to see the new Union Oil Research Center. Just completed last year at a cost of eight million dollars, the Research Center boasts some of the best modern facilities for research on the west coast. Dr. W. E. Bradley, Director of Research at the Center, welcomed the members and gave an introductory talk on the "Organization and operation of an industrial research group." Specially guided tours through the Center followed the talk.

STORING RUBBER TUBING

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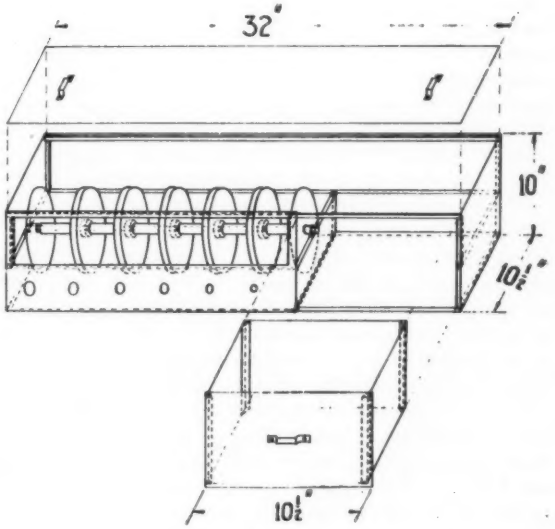
Diamond Research Laboratory, Johannesburg, South Africa

In most laboratories a stock of various sizes of rubber tubing is kept in a drawer or cupboard, or on a shelf. Unless an exceptional amount of storage space is available, the rolls of tubing as purchased from chemical supply houses must be stacked together and they soon become mixed and untidy. A convenient arrangement to store rubber tubing in a minimum space is illustrated below.

The different sizes of tubing are wound on a number of light reels, which have a slight frictional grip on a fixed shaft. The shaft may be installed in a cupboard or shelf. Spacers are provided between each reel so that they may rotate individually. If the reels turn too freely, the rubber tubing may uncoil itself. To overcome this, felt washers fixed to the shaft are placed between the reels. The reels can be made out of two pieces of $^3/_{16}$-in. hardboard 8 in. in diameter, fastened by screws to both ends of a $1^3/_4$-in.-diameter wooden center piece $2^1/_2$-in. long. The center piece is drilled with a $^3/_4$-in. hole to accommodate a brass rod serving as a shaft for the reels.

A brass staple in the wooden center piece holds one end of the tubing firmly while winding the latter on the reel. The free end of the tubing is drawn through a hole in a piece of hardboard fixed in front of the reels. The bore and wall thickness of each type of tubing are painted on the hardboard sheet, above or below the protruding end. Above the hardboard is fastened a transparent plastic sheet, to afford a full view of the reels of tubing. A small drawer can be provided as indicated to hold odds and ends of tubing, or short used lengths.



In this way a number of sizes of tubing can be stored neatly in a small space on a shelf or in a cupboard, readily accessible, and in full view for checking stocks. The device will be found useful in industrial laboratories and the chemical storerooms of educational institutions.

Report of the

NEW ENGLAND ASSOCIATION OF CHEMISTRY TEACHERS

CHEMICAL REACTIONS IN THE CORROSION OF METALS[1]

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THE rusting of iron, the most familiar example of metallic corrosion, has been associated in man's mind since earliest times with such undesirable and unlovely processes as corruption, pollution, and deterioration. The fact that a very substantial proportion of the iron and steel produced in the present century has reverted already by corrosion processes to oxides and salts portends the depletion of the world's most useful metal. In early colonial times, iron for local needs was mined on Staten Island in New York harbor. Northern New Jersey supplied much of the iron required in the Revolutionary War and for a century afterward. Then in the 1890's the rich Mesabi range of northeastern Minnesota became the chief source of iron ore for our expanding steel industry. With the approaching exhaustion of the Mesabi ores, tremendous and fascinating iron mining developments are being undertaken in Labrador and Venezuela. Some future generation, early in the next century, will be faced doubtless with the depletion of these new reserves.

In addition to the irreparable dissipation of natural resources, metallic corrosion represents an astounding financial loss. A recent careful survey indicates that the direct cost of corrosion including measures for retarding or minimizing corrosive action, such as the application of protective coatings, amounts to over five billion dollars annually in the United States alone.[2]

Corrosion is evident to everyone in everyday life. Kitchen equipment, hand tools, automobiles, domestic water heaters, wire fences—all metallic articles, except those of metals such as gold and platinum, are subject to corrosive attack in ordinary environments. The corrosion cost of automobile mufflers was 66 million dollars and domestic water heaters 225 million dollars in 1948, to cite two easily recognizable examples.[3]

The reactions of corrosion are concerned with the deterioration of the metallic state. Although unwanted, these reactions are both respectable and interesting. Broadly speaking, corrosion deals with the processes by means of which metals react with nonmetallic elements of their environments producing chemical compounds. The nature of these compounds will influence the course of corrosion. If highly insoluble and deposited as an impervious film in intimate contact with the metallic surface, the corrosion reaction may be retarded or stopped; if soluble or not precipitated on the surface, the rate of corrosive attack will not be inhibited; and, finally, if only part of the surface is covered with corrosion products, the action may be accelerated in the uncovered areas, producing pitting.[4]

The processes of corrosion may be viewed as electrolytic in character, that is, dependent upon the operation of electrolytic cells at the metal surface. In the direct oxidation or tarnish of metals in the absence of moisture or at higher temperatures, the corrosion cell consists of the metal surface acting as anode and a monomolecular layer of oxygen, sulfur, or other nonmetallic element in contact with the metal serving as cathode. In the action of this cell, both metallic ions and electrons migrate from the metal into the interfacial region where the electrons ionize the adsorbed nonmetallic elements, oxygen or sulfur, to anions. These in turn unite with the migrating cations to form a solid state lattice structure on the metal surface. The subsequent course of oxidation depends upon the physical structure and electrical resistance of this solid film. If film weight is plotted against exposure time, those metals which develop protective films of appreciable thickness, as for example aluminum and lead, become constant in weight with time; where the film is porous weight gain is a linear function as exemplified by the case of zinc; and, finally, where rate of attack depends upon a diffusion process, the weight gain characteristic is likely to take a parabolic form.

The more familiar type of corrosion reaction occurs

[1] Presented at the 14th Summer Conference of the NEACT at the University of Vermont, Burlington, Vermont, August, 1952.
[2] UHLIG, H. H., Proc. United Nations Scientific Conference on Conservation and Utilization of Resources, Vol. II, p. 213.
[3] UHLIG, H. H., *Chem. Eng. News*, **39**, 2764–7 (1949).
[4] BURNS, R. M., *Bell System Tech. J.*, **15**, 20–38 (1936).

in the presence of moisture and consists essentially in the corroding metal replacing hydrogen in the water molecule. In this process, two reactions occur simultaneously at different areas on the metal surface; that is, metal ions pass into solution at the anodes and hydrogen ions are discharged as atoms at the cathodes of electrolytic cells which arise as the result of heterogeneities at the interface between metal and environment. For example, the following are typical of the electrolytic cells by means of which corrosion may occur:

Anode	*Cathode*
Base metal	Noble metal (2 metallic phases)
Metal	Oxide
Metal	Carbon particles
Metal (absence of O_2)	Metal (presence of O_2)
Strained metal	Annealed metal

The anode reaction of these electrolytic cells in the corrosion of iron is:

$$Fe - 2e \rightarrow Fe^{++} \quad (1)$$

and at the cathode the reaction is:

$$2H^+ \text{ (from dissociation of water)} + 2e \rightarrow 2H \quad (2)$$

combining these reactions, the equation for the solution of the metal becomes:

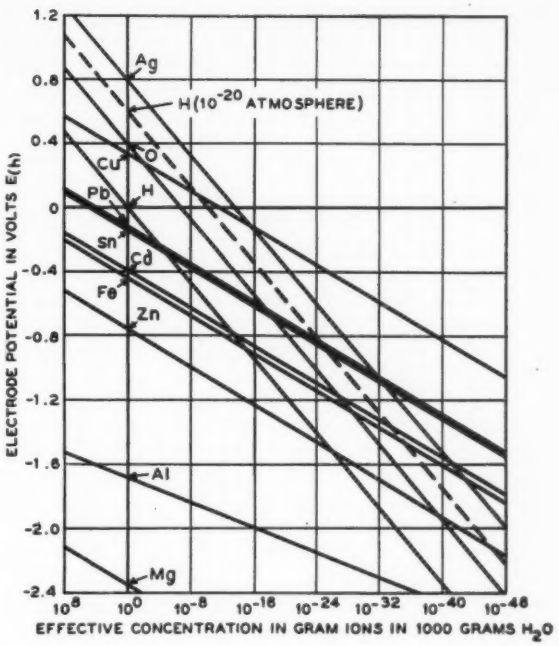
$$Fe + 2H^+ \rightarrow Fe^{++} + 2H \quad (3)$$

The atomic hydrogen discharged in equation (2) polarizes the cathodic areas and retards corrosive action unless removed. That is, since the potential of the corrosion cell is usually less than the hydrogen overvoltage of the metal, the rate of liberation of molecular hydrogen gas is very low. In the presence of oxygen, however, removal of hydrogen or depolarization of the cathode proceeds thus:

$$2H + {}^1/_2\, O_2 = H_2O \quad (4)$$

When the concentration of ferrous and hydroxyl ions exceeds the solubility product of ferrous hydroxide, this corrosion product will be precipitated usually at an appreciable distance from the metal surface. In the presence of oxygen, it will be partially or wholly converted to the less soluble ferric hydroxide. The resulting film of corrosion products is usually porous and of little protective value. If, however, strong oxidants, such as potassium dichromate, are supplied to the metal surface, a film of ferric hydroxide of more continuous character is produced in intimate contact with the iron surface and protects it from corrosion. At high concentrations of nitric acid, iron becomes passive.

The driving force or potential of corrosion cells may be approached from a consideration of the electromotive forces series. It will be recalled that this is an arrangement of the elements in order of their dissolution tendencies in solutions of their own ions of corresponding concentrations—one gram-ion in 1000 g. of water being taken as the concentration for basis of comparison. These familiar standard potentials of the elements may



be considered as the voltages of electrolytic cells in which the other pole is the hydrogen electrode at one atmosphere of hydrogen gas pressure in a solution containing one atomic weight in grams of hydrogen in 1000 g. of water at 25°C.—that is, the standard hydrogen electrode.[5]

It may be said at once that the electode potentials of practical corrosion cells deviate markedly from these standard values of the e. m. f. series. The standard ionic concentrations and hydrogen gas pressure are usually not provided by nature. It will be recalled, however, that every tenfold change in ionic concentration produces a change in electrode potential of $0.059/n$ volts at 25°C. where n is valence. If the change in potential of some of the common metals is plotted against change in ion concentration, it will be seen that the ordinate corresponding to a concentration of one gram ion provides the well-known e. m. f. series. Moreover, since the potential change of monovalent elements with ionic concentration is twofold that of divalent and threefold that of trivalent elements, it will be observed that the potential relationships of certain pairs of elements of different valences may be reversed in different concentration ranges. If a different ion concentration were chosen as basis of comparison, the order of the e. m. f. series would be modified. To consider a single pair or couple, monovalent silver and divalent copper may be chosen to illustrate that identical potentials occur at an ionic concentration of 10^{-16} gram ions per 1000 g. of water. This comparison assumes that each metal is in equilibrium with corresponding concentration of its own ions, a condition not usually realized in practical cases. Usually two metals exposed to a given environ-

[5] Burns, R. M., *ASTM Bull.*, No. 126, p. 17 (Jan., 1944).

ment will develop markedly different ion concentrations. Thus, the potential relationship of a pair of metals will depend in a large measure upon the nature of their environment and may undergo a reversal with change in surrounding medium. Students can illustrate this reversal in simple experiments with galvanic couples. For example, if iron-lead couples are immersed one in dilute acid and the other in dilute alkali solution, it will be found that the anodic or corroding member of the couple in acid will be iron and in the alkali it will be lead. In each case, the cathodic electrode may suffer minor local action attack. This reversal of potential induced by environmental change is understandable when it is known that the concentration of ferrous ions which develops upon the immersion of iron in acid solutions is approximately one gram ion per 1000 g. of water but only about 10^{-10} in alkaline solutions. On the other hand, lead ion concentration upon immersion of the metal in acid will be in the range of 10^{-3} to 10^{-5} gram ions per 1000 g. of water, and 10^{-24} or less in alkaline solutions. Inspection of the potentials of iron and lead corresponding to these ionic concentrations in the graph will illustrate this reversal.

It will be recalled that several examples of corrosion cells cited earlier do not involve contact of dissimilar metals. In such cells the cathodic element may be other areas of the corroding metal acting as hydrogen electrodes. As is well known, the potential of the hydrogen electrode depends upon the hydrogen gas pressure. Since this is usually far less than one atmosphere, the hydrogen electrode cathode in practical corrosion cells is more noble than the standard value. Indeed, at 10^{-20} atmosphere it becomes 0.58 volts electropositive (noble) to its value at one atmosphere, as shown in the graph. This is because tenfold hydrogen pressure changes result in a potential change of 29 millivolts. The actual hydrogen pressures of corrosion cell cathodes depend upon the relative rates of hydrogen discharge and removal which in turn depends upon cell potential and rate of cathode depolarization. If oxygen is present in excess of that required to combine with the liberated hydrogen, then the cathodic areas become in reality oxygen electrodes of still more noble potential.

Thus the extent of anode and cathode polarization of corrosion cells determines the rate of current flow and consequently the rate of corrosion. For example, a deficiency of oxygen or the presence of certain compounds such as thiourea, butyl sulfide, diethylamine, sodium silicate, etc., promotes cathode polarization. On the other hand, strong oxidants such as dichromates or salts which form highly insoluble compounds with the metal may induce passivation of anode areas and thus retard or prevent corrosion.

This discussion has been concerned chiefly with the corrosion of iron. Nonferrous metals corrode by analogous reactions with the difference that protective films are more readily formed. Corrosion-resistant alloys, such as stainless steels, owe their passivity to the ability of their nonferrous components to develop resistant films. In this case, the maintenance of passivity is usually dependent upon the presence of oxygen. Among film-destructive agents, the chloride ion is most effective.

The foregoing review of the chemical reactions of metallic corrosion outlines the rather simple chemistry that is involved when metals are exposed to complex natural environments. Perhaps it is now appropriate to speculate on the suitability of corrosion experiments and problems for arousing student interest in chemistry. Reference was made earlier to experiments designed to demonstrate the influence of environment upon the electrode potential relationship of a pair of metals. Other simple experiments could be suggested to illustrate the relative corrosiveness to different metals of various salt solutions, such as chlorides, nitrates, sulfates, sulfides, nitrites, carbonates, phosphates, silicates, etc. The influence of oxygen or hydrogen ion concentration on corrosion of iron can be tested readily. A practical problem that should interest students is that of determining how much sodium dichromate should be added to the radiator of the family car to protect it from corrosion; or how much of this compound is required to prevent corrosion of iron by a refrigerating brine containing 20 per cent calcium chloride.[6] Tin coatings on tin cans are porous yet little or no corrosion occurs inside the can even when not lacquered until it is opened to the moist atmosphere.[7] Why? Since this is more difficult than it first appears, it might be suggested to the student that he experiment with a tin-iron couple employing a stewed fruit or fruit juice as electrolyte and that he look up the subject of complex ions. Other problems that might be suggested are: (1) Why is lead corroded readily in distilled water but not in most soil waters?[8] (2) Why does aluminum protect iron in sea water but not in fresh water? (3) In view of the fact that copper is noble or cathodic to hydrogen in the electromotive force series, why does it dissolve in dilute nitric acid? (4) Devise experiments to show that copper-bearing natural waters corrode aluminum cooking utensils and explain the action.

If problems of this sort are of any value in stimulating student interest in natural science, it may be worth while to see whether the student's approach to the solution of such problems gives any indication of talent for scientific work. In view of the decreasing proportion of enrollments in science and the urgent need for more scientists, it is of the utmost importance that evidence of scientific talent in students be recognized, encouraged, and nurtured. Since 1900 the proportion of high school students taking courses in physics has fallen from about 19 per cent to 5.5 per cent[9]

[6] *Refrig. Eng.*, **14**, 173–82 (1927); ROETHELI, B. E., AND G. L. COX, *Ind. Eng. Chem.*, **23**, 1084–90 (1931); DARRIN, M., *Ind. Eng. Chem., Anal. Ed.*, **13**, 755–9 (1941).

[7] BURNS, R. M., AND A. E. SCHUH, "Protective Coatings for Metals," Reinhold Publishing Corp., New York, **1939**, pp. 156, 157.

[8] BURNS, R. M., *Bell System Tech. J.*, **15**, 603–25 (1936).

[9] DUBRIDGE, L. A., *Proc. Inst. Radio Engrs.*, **40**, 900 (1952).

and, while the record in chemistry is possibly better, the proportion has declined markedly. This is particularly serious in view of the vastly increasing demands for scientists and engineers. About 3300 industrial concerns have research programs[10] and their needs, together with those of government laboratories and universities, far exceed the supply of trained scientists either present or prospective. Another measure of expansion of scientific work is the sevenfold increase in expenditures which has taken place in the past 12 years.

Since national survival, as well as continued industrial progress, depends upon the maintenance of a high level of scientific effort, it seems essential that there be a wider understanding of the nature of science as a human enterprise and that every young person possessing potential talent for scientific work be discovered and attracted to the field.

[10] SCOTT, E. W., Editor, "Applied Research in the United States," Publication 210, National Research Council, Washington, D. C., 1952.

This Association and its members would seem to have an unusual opportunity to contribute significantly toward the goal of increasing public enlightenment and of discovering more prospective scientists. Originality, curiosity, imagination, objectivity, and creativeness are among the relatively rare qualities of mind that characterize the potential scientist. Students so gifted deserve the major proportion of their teachers' efforts; for teachers can make no greater contribution to present-day society than to aid effectively in the individual development of those who will constitute the aristocracy of scientific brains. For they are of the "Fifth Estate" so thoughtfully defined some years ago by A. D. Little as "those who have the simplicity to wonder, the ability to question, the power to generalize, the capacity to apply. It is in short," said he "the company of thinkers, workers, expounders, and practitioners upon whom the world is absolutely dependent for the preservation and advancement of that organized knowledge we call science."

• QUANTITATIVE ANALYSIS

Harvey Diehl, Professor of Chemistry, Iowa State College, and *G. Frederick Smith,* Professor of Chemistry, University of Illinois. John Wiley & Sons, Inc., New York, 1952. vii + 539 pp. 99 figs. 44 tables. 15.5 × 23 cm. $5.

THE subtitle, "Elementary Principles and Practices," describes this book rather well. The authors have attempted to "emphasize chemistry, paying as much attention to technique as to theory, and including only those items of theory of direct benefit to the working analyst." The reviewer has been very favorably impressed with the result. The treatment of theory is still at a very high level and the added attention to laboratory practices and technique should be of considerable value to students. The book has been written as a text for a year course at the sophomore level. No references have been given.

Colorimetric analysis has been given special emphasis in a separate chapter in recognition of its increasing importance. Gravimetric, volumetric, and colorimetric analysis are presented in that order. The authors mention that for several years they taught volumetric analysis before gravimetric analysis but finally returned to teaching gravimetric analysis first, with a considerable feeling of relief. Besides the procedures which are customarily found in an elementary text on quantitative analysis, a number of new ones have been included, such as the Versene method of determining hardness of water, determination of iron with bathophenanthroline, and standardization of permanganate with ferrous ethylenediammonium sulfate (other methods of standardization are also given), a modified Volhard procedure for bromide.

Theoretical treatment of oxidation-reduction has made use of the convention of reduction potentials. Through the electrochemical theory of oxidation-reduction an attempt has been made to correlate oxidation-reduction, electrodeposition, pH, and neutralization. For the sake of consistency in relating pH and neutralization to the reduction potential series the figures utilizing the pH scale have been inverted with 14 at the top and 1 at the bottom. Unfortunately the color changes of some acid-base indicators have not been inverted similarly in several of the figures and consequently are in error.

An entire chapter has been devoted to a thorough discussion of the various methods of effecting separations. The reviewer feels that this chapter is very excellent but would have been even better if the use of chromatography in analytical separations had been given a few words.

Some instruction in the working of quantitative problems is outlined in appropriate places throughout the text. The 450 problems at the ends of the various chapters offer a wide choice in the making of problem assignments. A supplementary problem book lists answers and solutions to the problems. The discussion of errors, accuracy, and precision seems to be ample for a book at this level.

After reading this book the reviewer has the feeling that his few unfavorable criticisms are only of a trivial nature and that the book as a whole is a very excellent one. Furthermore he

feels that it is somewhat more than just an elementary text and that in future years students who have used it will be taking it off the shelves to find answers to their problems about quantitative analysis.

REX J. ROBINSON

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SEATTLE, WASHINGTON

● QUANTITATIVE CHEMICAL ANALYSIS

Leicester F. Hamilton, **Professor of Analytical Chemistry, and** ***Stephen G. Simpson,*** **Associate Professor of Analytical Chemistry, Massachusetts Institute of Technology. Tenth edition. The Macmillan Co., New York, 1952. xvii + 529 pp. 70 figs. 16 tables. 15 × 21.5 cm. $4.50.**

THIS well written text is the tenth edition of one first published in 1897 under the authorship of Henry P. Talbot. In the present edition, numerous changes have been made but in general the authors have attempted to clarify presentation and to modernize and expand the theory. The reviewer is happy to note that the authors have adopted the "so-called Lewis convention" for the sign of electrode potentials. This is certainly a desirable change.

The text is divided into five main parts. Part one is an introduction which includes among other topics, The Analytical Balance, Presicison of Analytical Measurements, and Review of Some Basic Principles, particularly equilibrium; the four remaining parts consider in order, Volumetric Analysis, Gravimetric Analysis, Instrumental Methods, and Miscellaneous Determinations. Some of the miscellaneous determinations are sulfur in pyrite, analysis of plain carbon steel, and analysis of some commercial products. An appendix contains information on preparation of reagents, tables of values needed to solve problems in the text, and a brief discussion of the use of logarithms.

The text is a well balanced introduction to chemical analysis. There are many representative problems solved throughout the body of the text to illustrate the theory. These problems are solved in a clear, straightforward manner. At the end of practically all of the chapters there are thought-provoking groups of problems with answers, which furnish the student ample opportunity to test his mastery of the principles.

The laboratory determinations are familiar ones which are useful for their teaching value. They are not, in many cases, the latest or shortest methods for the analysis of a particular component but the reviewer believes that teaching value should take precedence in this matter. The general plan of presentation of a determination is the theory, a detailed description of the procedure, and representative calculations. The number of laboratory determinations offered to the instructor is sufficiently large to allow considerable freedom of choice.

More extensive discussions of determinate errors, organic precipitants, buffer solutions, and the calibration of volumetric glassware would be of value, particularly to the chemistry major. The calibration of the pipet and of the volumetric flask is treated quite briefly. A larger number of problems on calibration of volumetric glassware would be helpful to the student.

The text is relatively free of errors. Those noted are minor. For example, on page 379 the electron symbol, ϵ, is omitted from an equation, and on page 93 an exponent in one of the equilibrium constant expressions is incorrect. In the description of the analytical balance on page 49 there is a change in nomenclature of a balance part from "column" in the figure to "post" in the description. In Chapter XXIV Bouguer's law is credited to Lambert.

Where appropriate, ionic equations are used throughout the text. The authors digress from this practice occasionally, *e. g.*, hydrolysis of ammonium chloride on page 142.

Adherence to the recommendations of the committee on nomenclature, Division of Analytical Chemistry of the American Chemical Society, would eliminate the ambiguity of the first sentence on page 285. Analytical chemists could help promote better nomenclature practices by following the recommendations of the Committee of the International Union of Chemistry and thus eliminate such names as potassium bisulfate, sodium bicarbonate, and potassium bi-iodate. The outline on page 393 may be inclusive enough for the scope of this book, but it is not sufficiently inclusive to cover a number of different kinds of methods. An introduction pointing out the limitations of the outline would be in order.

The authors briefly consider the Brönsted-Lowry concept of acids, bases, and salts on page 91 and then point out that "the old definitions of acids, bases, and salts will therefore be retained in this text." However, in dealing with carbonates, hydrogen carbonates, and phosphates the protonic theory is used. Even though the definition of pH as given on page 128 may be adequate for a course in elementary quantitative analysis, a brief explanation of a more accurate definition would be informative for the student.

The paper, printing, and binding are of good grade and suited for practical use. In general, the figures are very good. Dimensional markings on the wash bottle in figure one would be helpful and, in addition, might reduce the space needed to describe its preparation.

In the reviewer's opinion this text is to be held in high regard and can be recommended to all teachers of elementary quantitative analysis for their careful consideration.

GLENN H. BROWN

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● PROBLEMS OF LIFE

Ludwig von Bertalanffy, **University of Ottawa, Late Professor of the University of Vienna. John Wiley & Sons, Inc., New York, 1952. xi + 216 pp. 14.5 × 22.5 cm. $4.**

IN WHAT might be called an essay on "general biology" in its broadest sense, the author discusses his concepts of life and organic processes as dynamic, open systems, culminating in his "general system" theory. A more elaborate treatment of this is planned for a following volume. The author makes it plain that he is not attempting to reduce biological processes to the terms of chemical and physical laws, but envisions them as higher levels of order and organization than are found in mechanical systems. Nevertheless, readers without a taste for metaphysics may find some parts of this essay in what we might call "metabiologics" a bit hard to follow, and will look for more specific details in the next installment.

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● PHYSICAL CHEMISTRY

Frank H. MacDougall, **Professor Emeritus of Physical Chemistry, University of Minnesota. Third edition. The Macmillan Co., New York, 1952. xi + 750 pp. 97 figs. 130 tables. 14.5 × 21.5 cm. $6.**

THIS is the latest revision of the book first published in 1936 and revised in 1943. The material on atomic structure has been revised to include a discussion of quantum mechanics and some of its applications. The section on nuclear reactions has been expanded.

The theoretical material in the chapter on chemical kinetics has been revised to include an expanded kinetic treatment and a discussion of the theory of absolute reaction rates.

The format and typography are both very good.

ARTHUR A. VERNON

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BOSTON, MASSACHUSETTS

● ORGANIC CHEMISTRY

E. E. Turner, Professor of Chemistry, and *Margaret M. Harris,* Lecturer in Chemistry, Bedford College, University of London. Longmans, Green and Co., New York, 1952. xi + 904 pp. 16 × 25.5 cm. $10.

THE present text represents an attempt to depict the high lights of the most important contemporary fields of organic chemistry, and simultaneously to present a sufficient background of elementary organic information to make the highlights understandable. The need for such an approach was felt by the authors in view of the rapidly expanding factual and theoretical frontiers of the science, and the book is intended by the authors as a "tourist guide" to the various localities of these frontiers. The text purports to be sufficiently comprehensive to lead an intelligent student from the elementary foundations of organic chemistry to the stage at which he is capable of deriving information from original research documents, as well as to function as a source of refresher information for the practicing organic chemist.

Subject matter presentation follows the customary arrangement. The first 19 chapters deal with aliphatic subjects, including carbohydrates, cycles, and terpenes. The following eight chapters deal with aromatic chemistry. The final chapters treat steroids, more advanced stereochemistry, free radicals, organometallics, heterocycles, vitamins, and alkaloids. Discussion of each topic follows the familiar pattern of functional group syntheses and reactions, structure, and configuration. Considerable emphasis is placed on the data of physical measurements, and up-to-dat conclusions derived therefrom are interspersed frequently theroughout the text. Explanations oforganic processes in electronic terms receive frequent attention, although such treatment is by no means a uniform practice in all parts of the text.

It seemed to your reviewer that the primary drawback to the book arises from the essentially inaccessible objective the authors had in mind when writing it. An attempt to lead the reader from the most elementary material up to advanced, speculative, and frequently controversial subject matter in each chapter is too much to ask for a single-volume text. Such an attempt occasionally forces the authors into somewhat ludicrous postures, for example, their need to explain (page 166) to the reader that "Ph stands for phenyl, C_6H_5, a hydrocarbon radical of the 'aromatic' series" during a sophisticated discussion in electronic terms of allylic rearrangements occurring during halogenation. In general the authors' difficult objective has led to the result, in the earlier chapters at least, that the most elementary material is apt to be found randomly and disconcertingly interspersed throughout otherwise advanced subject matter. Conversely, as for example in the introductory Chapter I, inadequately developed arguments of a highly advanced character clutter up what would otherwise be a straightforward and logical treatment of introductory material. The over-all outcome seems to be that most of the book would probably be completely incomprehensible to the reader who was previously unaware that "Ph stands for phenyl . . . .," while the more informed reader might be occasionally annoyed by the intrusion of so much, to him, superfluous elementary matter. In this connection the latter chapters of the book, by which point the reader presumably no longer has need for elementary explanations, stand out by contrast as better written and more logically developed treatments of their subjects.

The book is open to several other rather trivial points of criticism, points which might be serious mainly for students having insufficient background. Explanations of technique and theory behind physical measurements are absent or too briefly handled. Electronic mechanisms are somewhat glibly presented with little emphasis on the experimental facts supporting them and with insufficient mechanistic correlation among diverse reactions. References are not emphasized, but more recent work is referred to by author and year, so that the book is not a particularly convenient springboard to the original literature. Nomenclature is not systematically developed and is occasionally incorrect. Advanced concepts and their accompanying symbolisms are employed with perhaps too little preliminary explanation.

Such minor inadequacies will be unimportant for the more advanced student, who will find in the book a wealth of interesting factual and theoretical material of recent origin. With the exception of a very few spots the book is lucidly written in an alert style and the text is on the whole well supplemented with illustrative equations. The typography is readable and free of error, and the paper quality and binding are adequate. Disregarding the high purchase price and the inclusion of rather inadequately treated elementary material, the book can be highly recommended for the intermediate student of organic chemistry.

WILLIAM A. BONNER

OAK RIDGE, TENNESSEE

● THE ELEMENTS OF NUCLEAR REACTOR THEORY

Samuel Glasstone, Consultant, U. S. Atomic Energy Commission, and *Milton C. Edlund,* Physicist, Oak Ridge National Laboratory. D. Van Nostrand Co., Inc., New York, 1952. vii + 416 pp. 87 figs. 16 × 23.5 cm. $4.80.

"THE Elements of Nuclear Reactor Theory," according to its authors, is a textbook intended for study by physicists, engineers, and others who are being brought into the reactor program of the Atomic Energy Commission and who lack previous training in that special field. It seems to be well suited to that purpose. The first chapters are concerned with basic facts of nuclear science ranging from very simple aspects of nuclear structure and stability to a fairly extensive discussion of uranium fission induced by slow neutrons. The bulk of the book is a mathematical treatment of the production, motion, and consumption of neutrons in thermal reactors. The special terms used in this treatment are clearly defined. The mathematical steps are given in considerable detail. No previous knowledge of nuclear science is assumed, but the mathematics is not elementary.

This book will be useful as a reference for students or teachers who wish a brief survey of reactor theory. Few such are likely to work through it to the end, but by judicious skipping of the pages relatively more dense in mathematical equations it is possible to find many sections which can be read rather easily. Particularly interesting are the discussions of delayed neutrons and of xenon poisoning, and their effects on the control of reactors.

Finally, it is recommended as an excellent source book for the alumnus of the Manhattan Project who learned something of the subject when everything about it was considered secret, and who now has no idea what he is permitted to teach and what is still secret.

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BERKELEY, CALIFORNIA

● METAL DATA

Samuel L. Hoyt, Technical Adviser, Battelle Memorial Institute, Columbus, Ohio. Second edition. Reinhold Publishing Corp., New York, 1952. xiv + 526 pp. 18.5 × 26 cm. $10.

THIS is a revised and enlarged edition of "Metals and Alloys Data Book," published in 1943. The enlargement has been accomplished in part by increasing the number of tables and graphs from 400 to 700, but also by adding new data to former tables. The new data are frequently identified by literature citations that show dates as late as 1952. The important new developments in super alloys, heat treated steels, molybdenum, titanium, uranium, and zirconium have not been neglected.

In the preface to the second edition, the author claims that readers have found that the book gives ready and reliable answers to questions that come up daily in the practice of their professions. This is readily believed of metallurgists, but it probably

would not be true of most chemists. There is little effort made to define terms and abbreviations, to state conditions under which data were collected, to provide understandable headings in the tables or to interpret the data, to aid the nonmetallurgist in using the book. The uninitiated is rightly expected to turn to textbooks on metallurgy for the needed instruction. This book is a collection of tables, not a treatise.

On the other hand, Chapter 11, entitled Additional Metals, gives an excellent brief summary of many of the physical and mechanical properties of the less familiar chemical elements that chemists would have great difficulty to find so conveniently collected elsewhere. Another table that will be of use to the chemist is a long collection of data on the properties of the elements in Chapter 12. Melting points, however, are given only in degrees Fahrenheit. An extensive table of simple tests for identifying metals includes blowpipe tests. This last chapter also includes many useful conversion tables for units.

One glaring fault in the book is the Periodic Table that appears as Table 547. This is in the form of the obsolete short form, and still includes Ma instead of Tc for element 43, Cb for No. 41, Ab for No. 85, Fa for No. 87, and shows 94, 95, and 96 as Group VIII transitional elements, analogous, to Fe, Co, and Ni. An ineffective attempt has been made to rectify some of the errors by footnotes. It is recommended that this feeble table be replaced by a corrected, modern table in the next printing. Fortunately this lapse is not typical.

This excellent data book deserves a place on the reference shelf of any technical library. The comprehensive index makes the wealth of information readily accessible to those who have the background necessary to use it.

LAURENCE S. FOSTER

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● SIX-MEMBERED HETEROCYCLIC NITROGEN COMPOUNDS WITH FOUR CONDENSED RINGS

(Volume 2 in the series "The Chemistry of Heterocyclic Compounds," *Arnold Weissberger,* Consulting Editor), *C. F. H. Allen,* Eastman Kodak Co., in collaboration with *D. M. Burness, Jean V. Crawford, F. W. Spangler, Eleanor R. Webster,* and *C. V. Wilson.* Interscience Publishers, Inc., New York, 1951. xiii + 345 pp. 7 figs. 41 tables. 15.5 × 23.5 cm. $10 for single volume, $9 for subscribers to the series.

THIS monograph discusses the heterocyclic nitrogen analogues of the seven different ring systems resulting from the various possible fusions of four benzene rings. In each of the seven chapters one of these ring systems is considered. The scope and arrangement of each chapter is such that compounds with one heterocyclic nitrogen atom in various positions of the ring system are discussed first. Compounds with two, three, four, and five heterocyclic nitrogen atoms are then discussed whenever information concerning such compounds is available. All compounds in which there is a nitrogen atom common to two rings of a fused system are omitted in stated anticipation of a subsequent volume concerning such types.

Although the author of this volume is C. F. H. Allen, each chapter has apparently been written by one, two, or three of the collaborators and only for Chapter VI does Allen list his name as co-author.

The length of an individual chapter varies from 16 to 93 pages. The longest chapter concerns the azabenzanthrenes. Other chapters cover azanaphthacenes, azabenz[a] anthracenes, azabenzo[c]phenanthrenes, azachrysenes, azatriphenylenes, and azapyrenes. Much of the material in the various chapters has been summarized in frequent tables of compounds and their important properties. References have been collected at the end of each chapter. Apparently the patent literature has been covered carefully, because a large proportion of the 652 references concern patents.

In any extensive discussion of organic compounds with the complexity of those within the scope of this volume, nomenclature becomes very important. The authors have chosen to use the so-called "a" or "aza" system to name these compounds. For example, the name 1-azanaphthacene, is assigned to the compound which *Chemical Abstracts* prefers to name naphtho [2,3-g] quinoline. Another example is 6,7-diazabenz[a]anthracene, an alternate name for dibenzo[b,f]naphthyridine which is the choice of *Chemical Abstracts.* It is the opinion of this reviewer that a name in accord with the "aza" system is more easily and rapidly translated to a structural formula than a name in accord with the system which *Chemical Abstracts* favors. This should not be assumed to be an adverse criticism of the nomenclature preferred for these compounds by *Chemical Abstracts,* because the indexer is faced with complex problems which have apparently been better resolved with the naming system used by that abstracting journal. At the beginning of each section dealing with a different nucleus, the structural formula of the nucleus is given with the "aza" name used throughout that section. Other names including that preferred by *Chemical Abstracts* and the *Ring Index* number where assigned are also given at this point. These alternate names wil also be found in the index so that the section in this book dealing with a particular nucleus can easily be located.

There is ample justification for the publication of a volume concerned with six-membered heterocyclic nitrogen compounds with four condensed rings. Many of these compounds are of considerable interest as dyestuffs and it is not surprising that a group of chemists from the Eastman Kodak Company should be the authors of a compilation of these compounds and their chemistry. Further, many of the compounds within the scope of this volume have interesting physiological properties. For example, the index lists sixteen references to sections in the text concerning studies of the possible carcinogenic or antimalarial activity of certain of the compounds. Two groups of alkaloids are discussed in this volume. One of these is the chelidonium group with 5-azachrysene as the fundamental ring structure. The other is the aporphine group with 6-azabenzanthrene as the nucleus. The most important purpose this work will serve, however, is to stimulate further research in this particular branch of heterocyclic chemistry. Even a casual reading of a chapter of this book will indicate the many gaps in the knowledge of this subject. The author and his collaborators have done organic chemistry a favor in making available these excellent discussions.

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● HETEROCYCLIC COMPOUNDS, VOLUME II. FIVE AND SIX-MEMBERED POLYCYCLIC COMPOUNDS CONTAINING ONE O AND S ATOM

Edited by *Robert C. Elderfield,* Professor of Chemistry, Columbia University. John Wiley & Sons, Inc., New York, 1951. vii + 571 pp. 15.5 × 23 cm. $15.

THIS book is the second published volume of a series concerned with heterocyclic compounds. The first volume of this series discussed three-, four-, five-, and six-membered monocyclic compounds containing one oxygen, sulfur, and nitrogen heteroatom. The present volume considers polycyclic compounds with one oxygen or one sulfur heteroatom. It is proposed that polycyclic compounds with one nitrogen heteroatom and heterocyclic compounds with two or more heteroatoms be considered in future volumes. Apparently polycyclic compounds with three- or four-membered isocyclic or heterocyclic rings with one oxygen, sulfur, or nitrogen atom are so little known that they are not to be given a separate treatment, although in Volume I of this series, the chapter entitled Ethylene and Trimethylene Oxides includes a discussion of compounds with an ethylene oxide ring fused to a benzene ring or to certain polycyclic ring systems.

The material within the scope of Volume II of this series has been divided into 14 chapters. These are as follows: 1. Benzofuran and Its Derivatives (67 pages), 2. Isobenzofuran, Phthalan,

and Phthalide (55 pages), 3. Dibenzofuran (Diphenylene Oxide) (22 pages), 4. Thionaphthene (19 pages), 5. Dibenzothiophene (9 pages), 6. Coumarins (44 pages), 7. Isocoumarins (12 pages), 8. Chromones, Flavones, and Isoflavones (48 pages), 9. Chromenols, Chromenes, and Benzopyrylium Salts: The Anthocyanins (66 pages), 10. Chromanones, Flavanones, Chromanols, and Flavanols: Catechin, Brazilin, and Hematoxylin (50 pages), 11. Chromans (26 pages), 12. Xanthones, Xanthenes, Xanthydrols, and Xanthylium Salts (82 pages), 13. Fluorans, Fluoresceins, and Rhodamines (32 pages), and 14. Thiochromans and Related Compounds (18 pages).

Chapter 1 was written by R. C. Elderfield and V. B. Meyer, Chapter 2 by R. C. Elderfield, Chapter 3 by W. E. Parham, Chapters 4 and 5 by D. K. Fukushima, Chapters 6–13 by S. Wawzonek, and Chapter 14 by D. S. Tarbell. It will be noted that almost two-thirds of this volume has been written by one author. Inasmuch as Chapters 6 through 13 are concerned with polycyclic compounds, each containing a six-membered ring with one oxygen heteroatom, a much more uniform treatment of the many types of compounds discussed in these chapters has been accomplished than would have been possible if several different authors had been involved.

The number of pages in this book which are devoted to the heterocyclic sulfur compounds (46 pages) is much less than that allotted to the heterocyclic oxygen compounds (504 pages). This difference is due in part to the smaller amount of information available on the heterocyclic sulfur compounds within the scope of this volume and in part to the stated intentions of the editor to slight these compounds somewhat because their chemistry ". . . parallels to a considerable extent that of their oxygen analogs." Further, the discussion of thioindigo and related compounds is rather brief in anticipation of a more complete discussion of indigo in a future volume.

A large number of natural products fall within the scope of this volume. The coverage of the natural products within the particular chapters tends to be somewhat uneven. For example, Chapter 1 devotes more than 11 pages to a discussion of the investigations leading to the structure of usnic acid and omits even brief mention of the polyhydroxybenzalcoumaranones which are known to be present in the anthochlor pigment mixtures of certain plants. Only six pages in Chapter 11 are devoted to a discussion of the tocopherols, while discussions of brazilin and hematoxylin cover thirteen pages in Chapter 10. In Chapters 6–13, the natural products of known structure within the scope of each chapter are listed in table form, giving common name, systematic name or structural formula, and references to isolation and synthesis of each. Except in the case of the anthocyanins, relatively little information has been presented which would be of assistance in the isolation, purification, and identification of the natural products. To some chemists, this may be considered as a fault of the volume. It has obviously, however, not been the intention of the editor to make of this volume a monograph on these aspects of natural products. In spite of this fact, there is much of value in this volume for the chemist interested in naturally occurring polycyclic compounds with one heterocyclic oxygen atom. Emphasis has been placed on discussions of syntheses and reactions of the various types of compounds and on the interpretations of the various reactions in such a way that the properties of the individual natural products can be understood more easily.

Certain chapters (3, 6, 8, 10, 11, 12) contain brief comments concerning the physiological or pharmacological activity of the group of compounds in each of these chapters. These comments are apparently meant to be guides to the pertinent literature on the subject rather than as important sources of information.

A 21-page index is included in this volume. This index is arranged mainly according to individual compounds or groups of compounds. The compounds in the various tables of natural products are not included in the index unless they are also mentioned elsewhere in the text.

The editor, the individual authors, and publisher are to be complemented for the production of this excellent volume. Praise is due especially to S. Wawzonek for his major role as author. All students of the chemistry of heterocyclic compounds and related plant products should have access to this book although the high price makes it likely that relatively few of these will purchase the book.

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● ENCYCLOPEDIA OF SURFACE-ACTIVE AGENTS

J. P. Sisley, General Director, Technical Institute of Studies and Research on Fatty Matters, and General Secretary, Association of Chemists of the Textile Industry (both in France). Translated from the French and revised by _P. J. Wood,_ Technical Director, Royce Chemical Co. Chemical Publishing Co., Inc., New York, 1952. iv + 540 pp. 15.5 × 23.5 cm. $15.

The original French version appeared in 1949, the English translation in 1952. The first hundred pages or so cover a discussion of types of surface-active agents, general methods of preparation, representative products with formulas, and applications.

A chemical classification of surfactants takes up about five pages, with many subclasses under the general headings of (1) Anionic compounds, (2) Cationic, (3) Nonionic, (4) Mixtures of anion-active compounds, and (5) Mixtures of various products with soaps, sulfonated oils, and synthetic detergents. Some 2500 commercial products are listed by trade name and manufacturer only, arranged in the order of this chemical classification. These products are next listed in alphabetical order by trade name, with specific information about each, which varies in completeness, but which may give composition, class, physical form, properties, reaction, applications, and references. The translator has attempted to bring the book up to date by adding as an appendix some hundred-odd products in alphabetical order with the same type of information.

The products listed are manufactured in the following countries: In America—Argentina, Canada, the United States; in Europe—Austria, Belgium, England, France, Germany, Holland, Hungary, Italy, Poland, Scotland, Switzerland, and Yugoslavia; in Asia—Japan. The number of products from each is in general related to the development of the chemical industry of the country. Japan is an exception, having only 2 listed; Hungary has 2, Poland 2, and Germany about 50.

The information supplied is well presented. When used as a reference one may consult the table of contents, or class and alphabetical listings, as no index is included. For information on U. S. made products, other published sources supply more complete and more up-to-date material, such as "Surface Active Agents" by A. M. Schwartz and J. W. Perry for general information, and J. W. McCutcheon's 1952 list of commercial products for specific information.

The value of the book is its world-wide scope. For those interested in learning what surfactants are being made in other countries, it is a unique source.

CORNELIA T. SNELL

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New York, New York

● HYDROGEN ION CONCENTRATION

John E. Ricci, Professor of Chemistry, New York University. Princeton University Press, Princeton, New Jersey, 1952. xxxvi + 460 pp. Illustrated. 16 × 24.5 cm. $10.

According to the preface, the purpose of this book is to consider the problem of hydrogen ion concentration in general and in detail without depending upon any theories of the mechanism of ionization. Instead, it is supposed "to present a systematic derivation of interrelated formulas for calculations involving the hydrogen ion concentration in various aqueous solutions of almost any degree of complexity."

The author has achieved a remarkably thorough and in many ways novel presentation of his subject. In great detail and with many drawings, he has discussed the determination of ionization constants, the calculation of pH, buffers, "ionization fractions," various kinds of titration curves, feasibility of titration, and titration errors.

He has outlined the material systematically in the table of contents, which covers 13 pages. His list of symbols and abreviations runs to seven pages. Leafing through the book upon first examination of it, one receives the impression that it must average a dozen mathematical equations per page. Awed by the author's accomplishment, the reader will agree that the latter part of the purpose expressed in the preface has been realized.

Yet, although it is probably not important, one may doubt that it has been done without depending upon theories of the mechanism of ionization.

Moreover, the book might have been more readable if the author had been satisfied to use the generally accepted symbols rather than inventing so many of his own. For example, to designate the concentration of hydrogen ions, he uses a capital *H* in italic; to designate the activity of hydrogen ions, he uses a capital **H** in boldface. Such deviations from standard practice make the book more difficult to read than it should be.

Sometimes the book is verbose; occasionally it is obscure. The second sentence of the first paragraph is a typical example; it reads as follows: "The 'reaction' or condition of the pure solvent will be taken as the meaning of *neutrality*."

One may also regard the author's distinction between salts on the one hand and acids, bases, and ampolytes on the other hand as unjustified, artificial, and contrary to his statement of purpose in the preface. Experimentally, it is generally agreed that many salts are acidic in water and that many others are basic.

However, despite these minor points, the book is a remarkable contribution to the field. Probably it will remain the last word for many years to come.

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• INITIATION AND GROWTH OF EXPLOSION IN LIQUIDS AND SOLIDS

(Cambridge Monographs on Physics) *F. P. Bowden* and *A. D. Yoffe*. Cambridge University Press, New York, 1952. xii + 104 pp. 68 figs. 20 tables. 5 plates. 14.5 × 22.5 cm. $4.50

The initiation of an explosion is a complex and sometimes seemingly mysterious series of phenomena. The reason for the occurrence of an explosion is at times obscure and the resulting destruction may prevent retracing to the beginning of the process. The apparent lack of reproducibility and difficult techniques necessarily involved have not made this an inviting field of research for many scientists. Professor Bowden and his students have constituted one of the few groups who have worked in this field and they have done much to clarify the thinking about the explosion processes.

"Initiation and Growth of Explosions in Liquids and Solids," one of the series of Cambridge Monographs on Physics, is an exposition of a relatively simple idea concerning the beginning processes in a detonation. Most explosions are initiated, it contends, by the creation of a hot-spot within the explosive. This hot-spot may occur as a result of the friction between sliding surfaces, perhaps enhanced by a particle of grit, or by impact which may result in the adiabatic compression of an air bubble. Whatever the source of the hot-spot, if sufficient energy is concentrated at a point it will start an explosion. These hot-spots, which may be 10^{-5} to 10^{-3} cm. in diameter, provide a sufficient concentration of heat to ignite the explosive. The process pictured as following the ignition is essentially that of a van't Hoff thermal explosion. A considerable amount of relatively simple but elegant experimentation is described which has been developed to test this basic idea. The techniques described include high-speed photography as well as a very elementary description of the use of the image converter tube for photography.

Throughout all this work Bowden and his co-workers have used only minute quantities of explosives. Hence they have not studied the steady-state detonation which occurs in bulk explosives. They assume, probably correctly, that the mechanisms which they have studied are the initial steps in a large-scale explosion.

The principal chapters of this short book are concerned with friction initiation, impact initiation in both liquids and solids, and the growth of the explosion to full-scale detonation. It is filled with pictures and diagrams and its style is particularly readable. Since, as is pointed out, the incidence of explosion is to some degree erratic, it is to be regretted that the authors have not always used as sound a statistical approach to this work as might be desired. The sample is often too small to be conclusive.

On the whole this is an excellent book which adds much to a field in which the available literature is entirely too limited. In fact the bibliography at the end, which contains less than 100 references, constitutes a good list (particularly of British work) of the unclassified publications in this field.

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• ELEMENTS OF FOOD ENGINEERING. VOLUME I

Milton E. Parker, Consulting Food Engineer, Director and Professor, Food Engineering, Illinois Institute of Technology, Chicago, with the collaboration of _Ellery H. Harvey,_ Professor of Food Engineering, Illinois Institute of Technology, and _E. S. Stateler,_ Consultant on Foods, Wahl-Henius Institute and Professorial Lecturer in Food Engineering, Illinois Institute of Technology. Reinhold Publishing Corp., New York, 1952. ix + 386 pp. Illustrated. 16 × 23.5 cm. $8.75.

This first of three projected volumes proposes a definition of food engineering and presents classes of unit operations and unit processes as they apply specifically to food processing. Most chemical engineers and mechanical engineers in the food industry may find that this new professional description of food engineer fits them more aptly. However, beyond the first chapter entitled Engineering Factors in Food Processing, the authors become almost completely concerned with qualitative descriptions of food processing and exclude almost entirely any engineering aspects. Perhaps such treatment is attributable to the infancy of food engineering. Or the authors may be reserving for the later volumes the principles of process design and equipment.

As general technology preliminary to food engineering, this volume is refreshing, interesting, and pictorial; it is alive with names of processing people, novel classifications, definitions, tables of standards, and statistics. The beginner student should find this book engrossing and stimulating. The seasoned food technologist will recognize numerous advances in processing technology although he is likely to find the treatment of his own specialty less than authoritative and sometimes vague. It is not atypical of the industry that when direct knowledge and familiarity are lacking, one must often depend upon sources that are incomplete or which emphasize unduly certain points of view, leading to an imbalance in presentation.

The early chapters are fascinating treatises dealing with nutritional aspects, and economic and processing factors of food production. Thereafter the function of this volume becomes that of describing the processing of "refined foods." These include the headings: wheat and corn flours; rice and oats milling; minor cereal flours; fats and oils; sugars, sirups, starches, and gums; food protein derivatives; spices, essential oils, condiments, and flavoring extracts; beverages and fermentation products; and nuts.

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NEWS

The Structure of Matter Series

Just four years ago, Wiley launched a new series to include outstanding works concerned with structural phenomena in matter. The series was aptly called the STRUCTURE OF MATTER SERIES, and Dr. Maria Goeppert Mayer of the University of Chicago was appointed advisory editor. Written by outstanding research workers, these advanced level books are intended to point the way to important applications of new methods or concepts.

This month marks the publication of the newest addition to the series, MICROWAVE SPECTROSCOPY, written at Duke University by Walter Gordy, William V. Smith, and Ralph Trambarulo. This first book in the field summarizes research information and provides the key to an easy familiarity with the instruments and experimental techniques of microwave spectroscopy. It gives a detailed description of significant spectrographic components and supplies the fundamental theory involved in the detection of microwave absorption lines. 1953. 446 pages. $8.00.

The first book published in the series was Rice and Teller's THE STRUCTURE OF MATTER. An important account of the properties and behavior of atoms, "Rice and Teller" set the standard for the other books in the series. Professor John A. Wheeler of Princeton University summarized the opinions of other reviewers when he wrote, "It will fill an important place in the scientific literature now occupied by no other book." 1949. 361 pages. $5.50.

Second in the series is Leverenz's AN INTRODUCTION TO LUMINESCENCE OF SOLIDS. Of this work the *Del-Chem Bulletin* said, ". . . a remarkable book which should satisfy even the most critical expert in this field; yet the inexperienced reader will find himself readily absorbing the detailed, but systematically presented material . . . should be invaluable both as a guide and text to those interested in this field." 1950. 569 pages. $12.00.

The third publication in the series, London's SUPERFLUIDS, Volume I: Macroscopic Theory of Superconductivity, drew this comment from *Nature:* "It can certainly be recommended to any research student embarking on the theoretical or experimental study of low-temperature phenomena." 1950. 161 pages. $5.75. Volume II is now in preparation.

Due early next year is THE MOLECULAR THEORY OF GASES AND LIQUIDS by Hirschfelder, Curtiss, Bird, and Spotz. It will treat equilibrium and non-equilibrium statistical mechanics in a unified fashion, discussing both the fundamental theory and the applications to practical physico-chemical and engineering problems.

WILEY BOOKS

NEWS of IMPORTANT TITLES

. . . *about* Wagner and Zook's **SYNTHETIC ORGANIC CHEMISTRY,** a new book designed to save hours of needless searching through scattered chemical literature. It places at your fingertips over 550 summaries of methods employed in synthetic organic chemistry—organized according to functional groups, familiar and readily accessible to all chemists. It gives you over 7000 key references to the chemical literature, including review articles, digests, and well-written experimental procedures. In addition, over 6000 organic compounds are conveniently tabulated. Here, summarized in a single volume, are the methods most frequently used in the preparation of monofunctional and difunctional compounds. *1953. 887 pages. $11.50.*

. . . *about* Samuelson's **ION EXCHANGERS IN ANALYTICAL CHEMISTRY,** the first book to thoroughly explain this rapid and simple method. Professor Samuelson, long known for his work in this field, presents a comprehensive treatment of the ion exchange method. One reviewer comments, "This is a well-conceived, clearly written, and adequately documented treatment of a technique which is gaining in importance and appreciation among chemists of all types. It presents systematically a variety of problems which can be solved by ion exchange methods. I consider the book valuable to practically every experimental chemist and recommend it strongly." *1953. 291 pages. $6.50.*

. . . *about* Hillebrand, Lundell, Bright, and Hoffman's second edition of **APPLIED INORGANIC ANALYSIS:** With Special Reference to the Analysis of Metals, Minerals, and Rocks. ". . . Messrs. Bright and Hoffman have done a great service to all who are concerned with inorganic analysis in revising Dr. Lundell's classic," wrote Mr. Robert M. Fowler, Union Carbide & Carbon Corporation. Professor A. P. Black of the University of Florida commented, "It should be in the hands of every chemist whose work carries him into the field of chemical analysis." Mr. H. A. Liebhafsky, General Electric Company, adds, ". . . it is a worthy successor to the first edition. Higher praise is scarcely possible." *1953. 1034 pages. $15.00.*

. . . *about the late* Earl K. Fischer's **COLLOIDAL DISPERSIONS**—*Chemical Engineering Progress* reports, "Dr. Fischer does an excellent job in the book. He does not spend too much time on theory or speculative ideas. He states briefly what the considerations are in each case and then goes on to the practical aspects." Dr. R. E. Allen of Rutgers University adds, "Dr. Fischer has accomplished the really formidable task of covering this complex and confused field, keeping the treatment brief without sacrificing clarity." *1950. 387 pages. $7.50.*

. . . *about* Audrieth and Ogg's **THE CHEMISTRY OF HYDRAZINE**—*Journal of the American Chemical Society* called it, ". . . a valuable and time-saving contribution . . . a veritable gold mine." Walter C. Schumb of M.I.T. added in the *Journal of Chemical Education,* "Altogether, it may be said that this book represents a compact, well-arranged, and authoritative account of the chemistry of this valuable substance, the utilization of which is perhaps only beginning to emerge from obscurity. It will be a welcome addition to inorganic chemical literature." *1951. 244 pages. $5.00.*

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Out of the Editor's Basket

New Extraction Apparatus

Scientific Glass Apparatus Co., Inc., is now manufacturing a new type of glass apparatus for the extraction and determination of salts of organic bases and acids known as the Schmall Apparatus.

According to the manufacturer, the technique employed eliminates many hand operations. Instead of the conventional method which involves a number of individual extractions of the liberated base or acid with immiscible solvents using separators, followed by titration in aqueous medium, etc., this new procedure not only facilitates sample preparation, but extraction is automatic, washings are eliminated, and labor cost of analysis reduced. When used in conjunction with titrations in non-aqueous solvents, transfers are eliminated and sharper end points obtained.

Two types of liquid-liquid extractors are available. One is for use with solvents lighter than water; the other for those heavier than water. For further information, write directly to the Scientific Glass Apparatus Co., Inc., Bloomfield, New Jersey.

"You Be The Judge"

One of our readers, Professor C. R. Naeser of George Washington University, has written us about an item which appeared in our eminent contemporary, the *Saturday Evening Post*, under their title "You Be The Judge," which we quote below. He points out that if this court decision is authentic it may be the basis of some trouble for instructors in freshman laboratories. An instructor who has to check what each student has weighed out may find himself very busy.

Readers will, of course, wonder what kind of a laboratory manual it could be which directs students to heat phosphorus and potassium chlorate in the same tube.

"Azalea and her sophomore classmates at Ivy Hall Female Seminary went to chemistry lab one afternoon to find the professor in a flurry. 'Girls,' she said, 'you each have a manual giving details of the experiment I want you to perform. As I have an appointment, I won't be here to supervise. So be sure to follow the directions exactly.'

"The professor thereupon rushed off, and the girls proceeded with the experiment. It involved combining ferric oxide and red phosphorus with potassium chlorate, and heating the mixture over a Bunsen burner. The manual warned that the experiment was dangerous unless the chemicals were mixed precisely in the proportions specified. Unfortunately, Azalea was too liberal with the red phosphorus. When she heated her mixture, it exploded and blew her into the nearest hospital. She subsequently sued Ivy Hall for damages.

" 'The professor had no right to assume that second-year chemistry students could follow the manual exactly,' she said. 'She should have stayed in the lab to check up. Her failure to do so was negligence, for which the school is responsible.'

" 'A college girl should be able to follow simple directions,' attorneys for Ivy Hall countered. "Even if the professor had been there, Azalea probably would have made the same mistake. Or did she expect the professor to mix each girl's batch?'

"If you were the judge, would you exonerate Ivy Hall Female Seminary?"

*****

"Azalea won. One of the three judges who heard the case ruled for Ivy Hall, but the two others decided that the professor should not only have remained with the class but should have checked each student's preparation before it was heated."

(*Based upon a 1941 decision of the United States Circuit Court of appeals.*)

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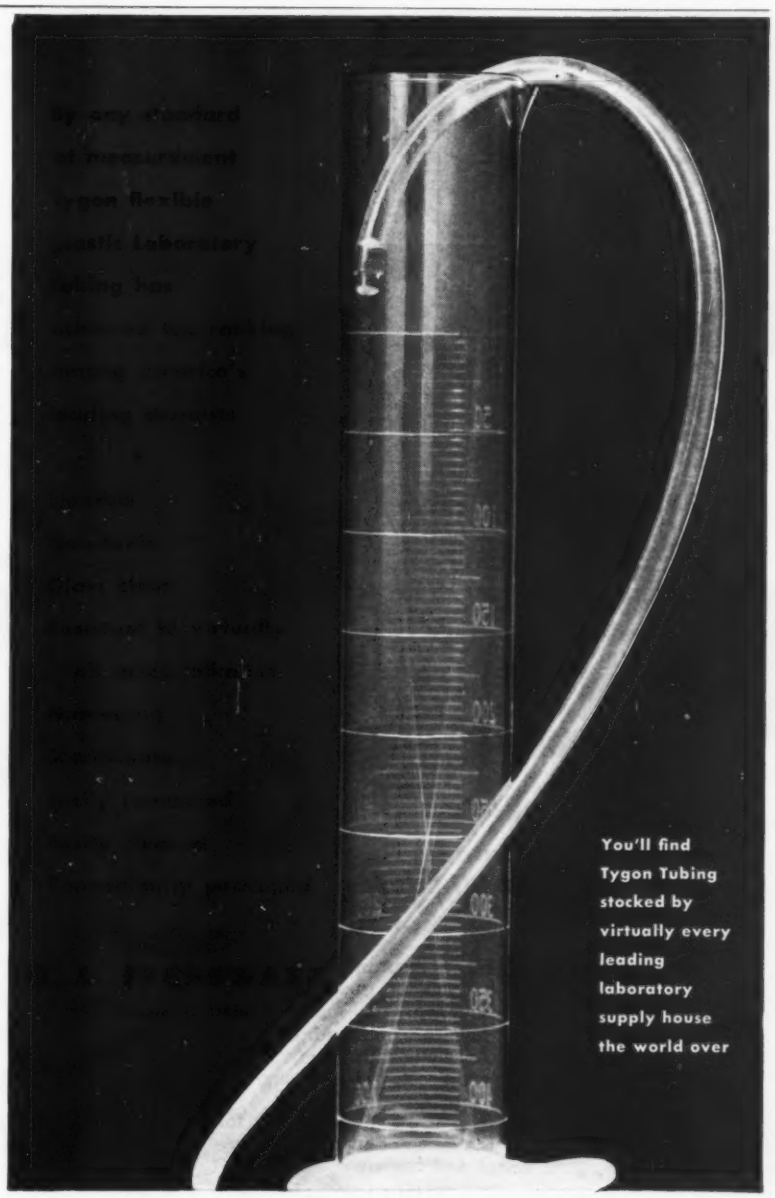

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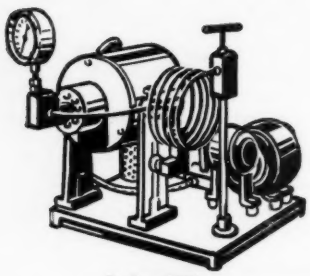

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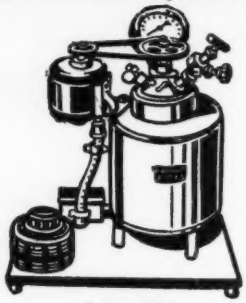
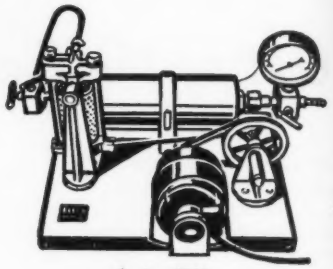


Series 4500

Medium Pressure -- Stirrer Type

For catalytic hydrogenation, alkylation, polymerization, and many other reactions requiring an autoclave with stirrer for pressures to 1000 psig. Furnished with either 1000 ml. or 2000 ml. stainless steel bombs, both interchangeable in the same electric heater for temperatures to 350° C.



Series 3910

Low Pressure -- Shaker Type

For catalytic hydrogenation and other reactions at pressures to 5 atm. Sturdy clamping device holds 500 ml. reaction bottle. Connections to 4-liter gas tank permit quantitative control of hydrogen consumed. Bottle heater available for temperatures to 100° Centigrade.

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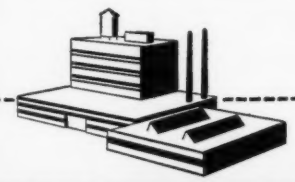

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cards, under the trade-name "Humigraph." The basic card contains seven indicator spots whose color changes from blue to lavender to pink under atmospheric humidity changes. The card is scaled to show relative humidity by reference to the color change spots. Applications are widespread in process plant and laboratory work, where quick and simple indications of humidity are essential. Detailed literature and sample cards are available at no cost.

New Cartesian Manostat

Completely redesigned to fulfill today's greater requirements of pressure control in the laboratory, pilot plant, and industry, the Cartesian Manostat No. 8 has recently been introduced by The Emil Greiner Co., 20–26 North Moore Street. New York 13, New York. This new manostat replaces the Emil Greiner Cartesian Manostat No. 5.

Based on the principle of the Cartesian Diver, Model No. 8 is greatly simplified in its operation. This self-contained unit has only to be placed in the system, set, and it functions without any supervision. It has an anodized aluminum body. A further improvement is the replacement of the stopcock with a needle valve by-pass.

It has high sensitivity, high capacity, and is corrosion resistant. Complete information on Model No. 8 and other Manostats is available in a recently published bulletin issued by the manufacturer.

New Literature

● *Catalog Number 100,* Section Two, giving complete details and prices of their newly redesigned line of incubators, paraffin ovens, and room incubators, is now available from Chicago Surgical & Electrical Company, 217 North Desplaines Street, Chicago 6, Illinois.

● A folder recently issued by Micro Metallic Corporation, Glen Cove, New York, illustrates and describes their complete line of porous stainless steel filters. Copies are available on request to the company.

● A new edition of their general *Bulletin Number 100* is now available from Photovolt Corporation, 95 Madison Avenue, New York 16, New York. This bulletin contains brief descriptions on all photoelectric and electronic measuring instruments manufactured by Photovolt.

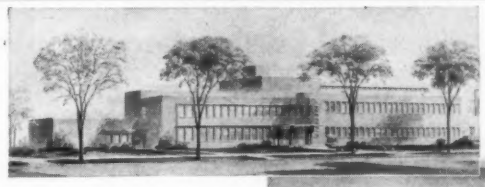
● An elaborately designed catalog of the complete line of Ohaus balances has just been published by Ohaus Scale Corporation, 1050 Commerce Avenue, Union, New Jersey. This 52-page booklet illustrates and describes Ohaus balances and weights and gives detailed construction features of these products. Copies are available on request from the company.

● A 16-page bulletin presents complete design and technical specifications on Boron Trifluoride Counters, Flow Counters, Linear Amplifiers, Scalers, Nucleometer, and radiation safety devices. Each instrument is illustrated and chassis views of electronic circuits are presented. Current price list on all RCL standard products is included. Copies of this bulletin may be had by writing Radiation Counter Laboratories, Inc., 5122 West Grove Street, Skokie, Illinois.

● The new Wakefield Industries *Catalog No. 22* reflects the expansion which has taken place in this company during the past year. This catalog is divided into three sections: Section I, Standard Scientific Glass Apparatus; Section II, Special Scientific Glass Apparatus; Section III, Scintillation Crystals and Liquids, Special Chemicals Gases and Accessories, and Thin Films. Also included are copies of the convenient Wakefield Industries "Sketch Sheet" for inquiries on special noncatalog scientific glass apparatus. Prices, descriptions, and illustrations of some 110 items, as well as photographs of Wakefield Industries personnel at work in their modern laboratory, are included in this attractive booklet. Write Wakefield Industries, Inc., 5108 West Grove Street, Skokie, Illinois, for a copy of this catalog.

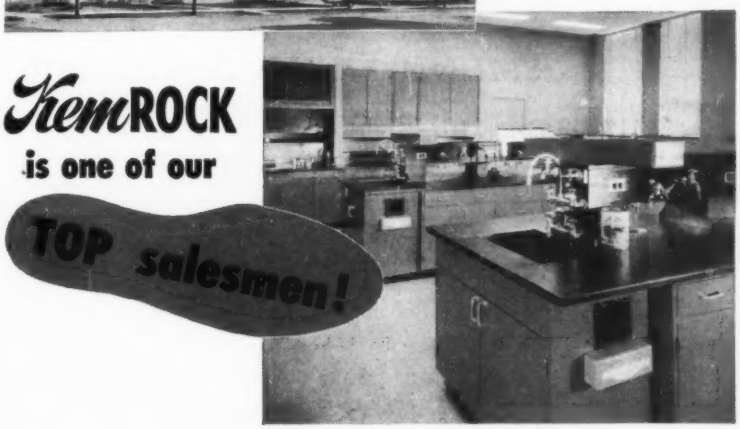
● A new *Catalog M,* just released by Nuclear Instrument & Chemical Corp., 229 West Erie Street, Chicago, Illinois, includes many new laboratory instruments—including a new sample changer, new medical counting system, new personnel monitoring systems, and other new equipment for detection and measurement of radioactivity. This catalog will be of interest to research workers in many scientific fields, and is available on request.

● Ten types of infrared absorption cell accessories are described in a new illustrated *Product Bulletin A-3,* just published by The Perkin-Elmer Corporation, Norwalk, Conn. These cells are used for handling solid, liquid, and gas samples during infrared analysis and their wide range greatly extends the usefulness of The Perkin-Elmer Infrared Spectrometers.



New Research Building of Wyandotte Chemicals Corporation, Wyandotte, Michigan.

One of Wyandotte's Laboratories showing Kewaunee Equipment with KemROCK Tops.



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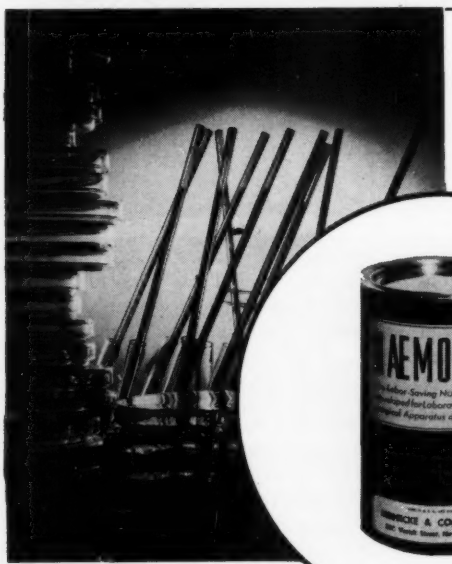

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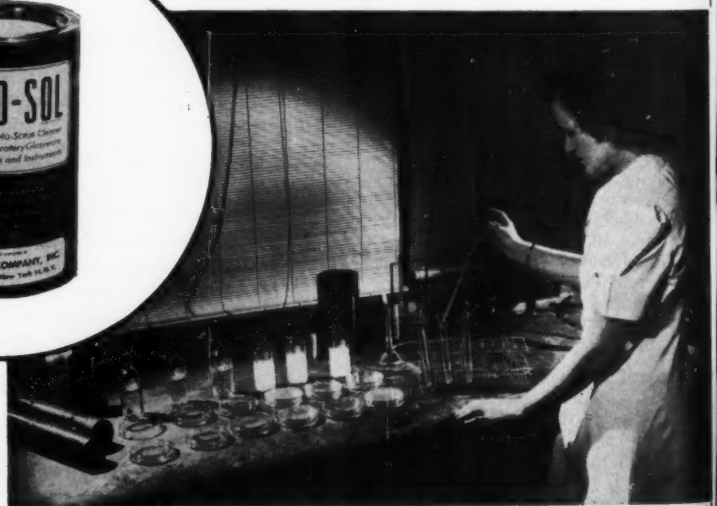

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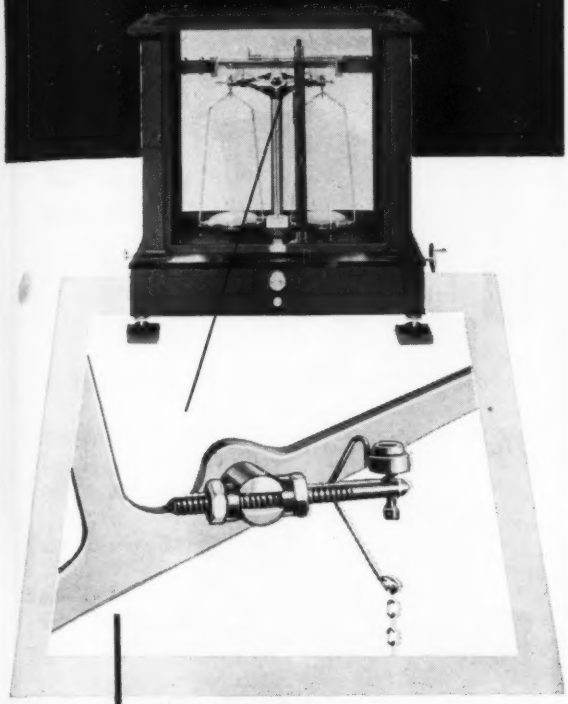

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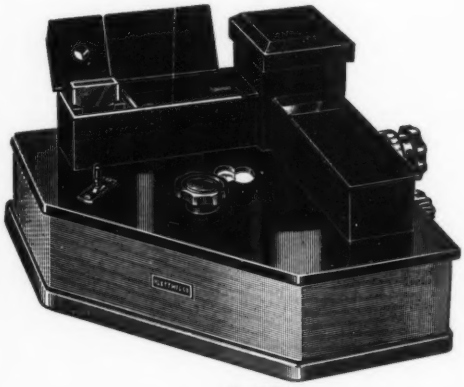

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References

(1) Ind. Eng. Chem. 25-653 (June, 1933)

(2) Ind. Eng. Chem. 25-1112 (Oct., 1933)

(3) National Bureau of Standards Journal of Research 12-241 (Feb. 1934, R.P. No. 649)

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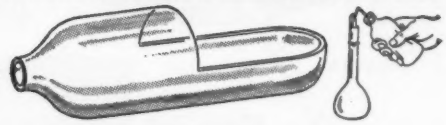

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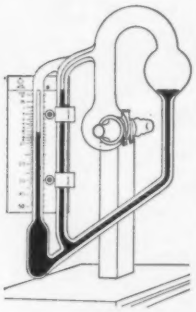

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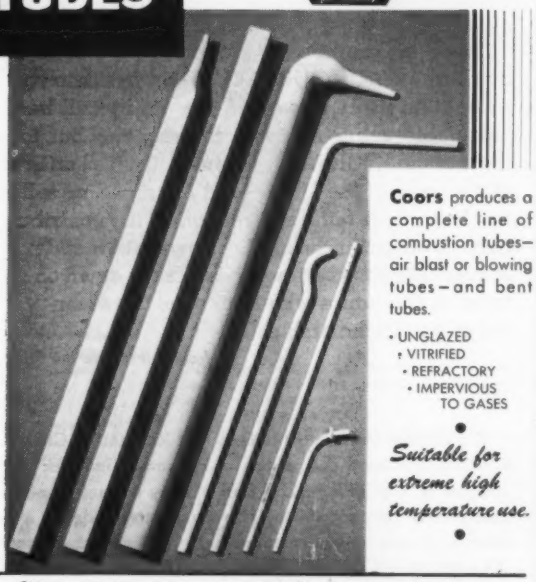

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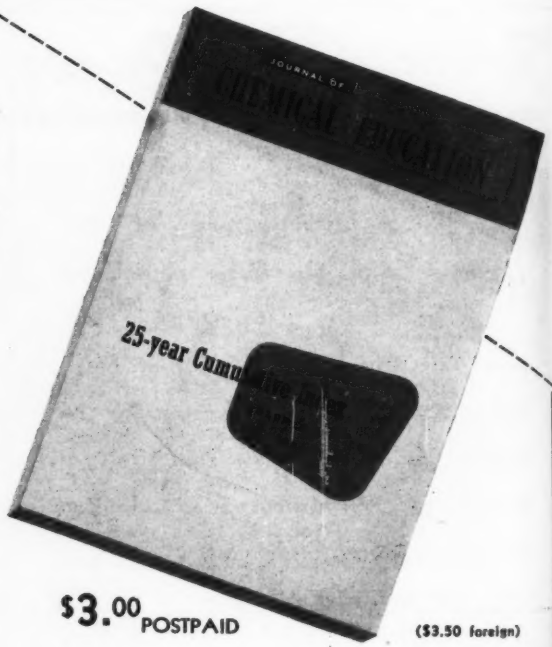

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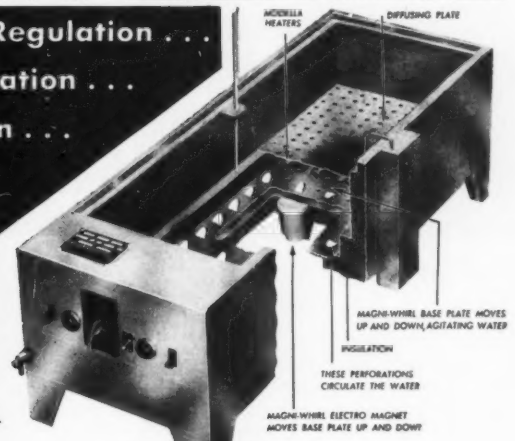
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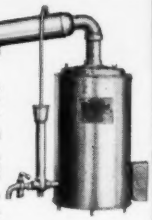

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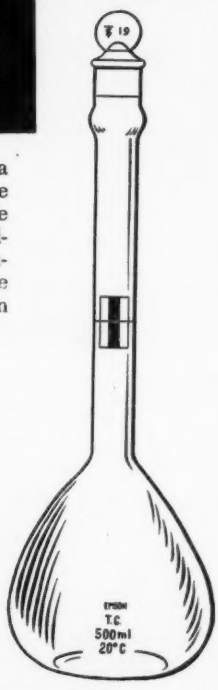

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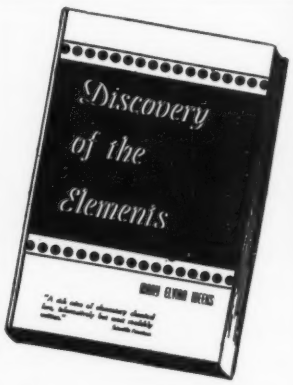

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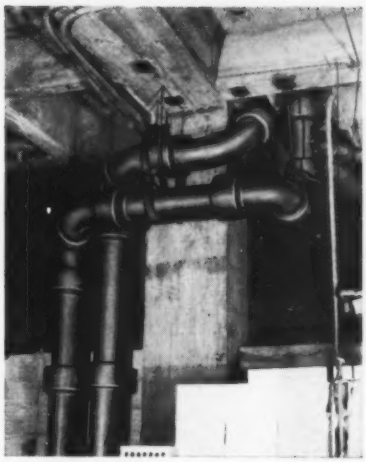

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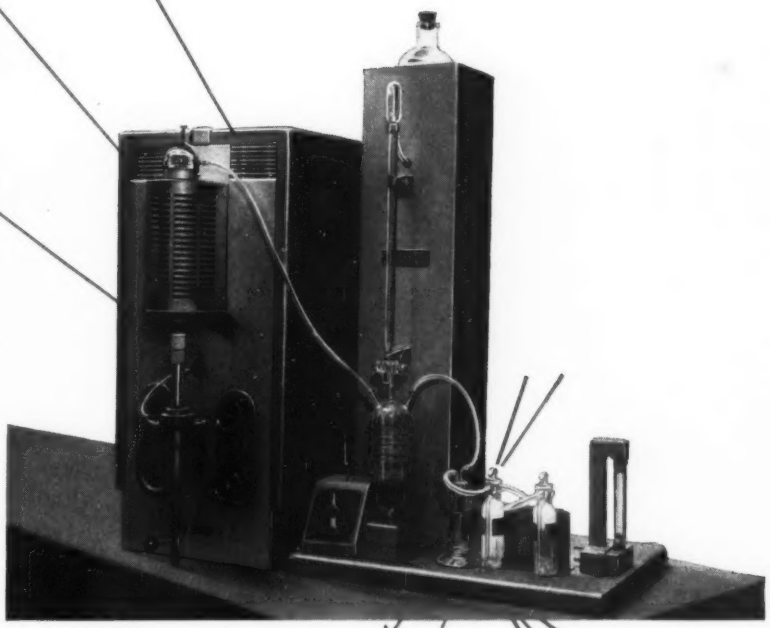

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